British Standard

BRITISH STANDARD

BS 1377-3: 1990

Incorporating Amendment No. 1

Methods of test for

Soils for civil engineering purposes —

Part 3: Chemical and electro-chemical tests

Committees responsible for this British Standard

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British Civil Engineering Test Equipment Manufacturers' Association
County Surveyors' Society
Department of the Environment (Property Services Agency)
Department of the Environment (Building Research Establishment)
Department of Transport
Department of Transport (Transport and Road Research Laboratory)
Coopted members

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Contents

Foreword

This Part of BS 1377 has been prepared under the direction of the Road Engineering Standards Policy Committee. It is a revision of clause **3** of BS 1377:1975 which is superseded by amendment. BS 1377 was first published in 1948 and first appeared in metric form in 1975.

BS 1377:1975 which has now been withdrawn is replaced by the following Parts of BS 1377:1990:

— *Part 1: General requirements and sample preparation;*

— *Part 2: Classification tests;*

— *Part 3: Chemical and electro-chemical tests;*

— *Part 4: Compaction-related tests;*

— *Part 5: Compressibility, permeability and durability tests;*

— *Part 6: Consolidation and permeability tests in hydraulic cells and with pore pressure measurement;*

— *Part 7: Shear strength tests (total stress);*

— *Part 8: Shear strength tests (effective stress);*

— *Part 9: In-situ tests.*

Reference should be made to Part 1 for further information about each of the Parts.

In this Part of BS 1377 the test methods described in the 1975 edition have been retained with some modification, and the following methods of test have been introduced:

a) determination of loss on ignition;

b) determination of carbonate content;

c) determination of chloride content;

d) determination of total dissolved solids content;

e) determination of electrical resistivity;

f) determination of the redox potential;

Sample test forms are shown in Appendix A.

It has been assumed in the drafting of this British Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

CAUTION. Attention should be given to the potentially hazardous nature of certain chemicals by following recognized practice and taking the appropriate precautions. These include the use of a fume cupboard when using acids and other toxic chemicals.

Special care and precautions may be necessary when dealing with contaminated ground (see DD 175).

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 42, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope

This Part of BS 1377 describes test methods for determining the amount of chemical substances, including organic matter, in samples of soil and ground water. The determination of some electrochemical and corrosivity properties of soil and water samples is also included.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purposes of this Part of BS 1377, the definitions given in BS 1377-1 apply together with the following.

2.1
titration

the addition of a solution from a graduated burette to a known volume of a second solution, until the chemical reaction between the two is completed. If the strength of one of the solutions is known, that of the other can be calculated from the volume of liquid added

2.2
indicator

a substance which is capable of giving a clear visual indication of the completion of a chemical reaction in a solution being titrated, usually by means of a change in colour

2.3
pH value

the logarithm to base 10 of the reciprocal of the concentration of hydrogen ions in an aqueous solution. It provides a measure of the acidity or alkalinity of the solution on a scale reading from 0 to 14, on which 7 represents neutrality

2.4
resistivity (of soil)

the electrical resistance, in ohms per unit length, of a column of soil of unit area of cross section. In this standard resistivity is expressed in ohm metres

NOTE This is to conform with SI units. The customary unit, $\Omega \cdot$cm, is equal to 0.01 $\Omega \cdot$m.

2.5
redox potential (reduction/oxidation potential)

a relative measure, expressed in millivolts, of the reducing or oxidizing capacity of soil, usually increasing with increasing oxygen content

3 Determination of the organic matter content

3.1 General

This procedure covers the determination of the percentage by dry mass of organic matter present in a soil. The method, which uses dichromate oxidation, is known as Walkley and Black's method.

NOTE Good practice in chemical testing requires that duplicate specimens should be tested. In each of the test methods the measurement of only one value of overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a mean value and an indication of the reliability of the results. Guidance on the number of measurements required and the treatment of the results obtained is beyond the scope of this Part of BS 1377.

Soils containing sulphides or chlorides have been found to give high results by this procedure. Methods for checking for the presence of these compounds, and procedures for their removal before testing if they are present, are included.

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

3.2 Apparatus

3.2.1 *Drying oven,* capable of maintaining a temperature of 50 ± 2.5 °C.

3.2.2 *Balance,* readable to 1 g.

3.2.3 *Balance*, readable to 0.001 g.

3.2.4 1 L *volumetric flask.*

3.2.5 *Two* 25 mL *burettes,* graduated to 0.1 mL.

3.2.6 10 mL *pipette and a* 1 mL *pipette*, each fitted with a rubber teat.

3.2.7 *Two conical flasks of* 500 mL *capacity.*

3.2.8 200 mL *and a* 20 mL *graduated measuring cylinders.*

3.2.9 *Desiccator,* (a convenient size is about 200 mm to 250 mm in diameter) containing anhydrous silica gel.

3.2.10 *Glass weighing bottle,* approximately 25 mm in diameter, 50 mm high and fitted with a ground glass stopper.

3.2.11 *Test sieves,* 2 mm and 425 μm aperture sizes, with receiver.

3.2.12 *Sample dividers of multiple-slot type (riffle boxes),* having widths of opening of 7 mm and 15 mm

3.2.13 *Pestle and mortar,* or a suitable mechanical crusher.

3.2.14 *Wash bottle,* preferably made of plastics, containing distilled water.

3.2.15 *Glass boiling tube.*

3.2.16 *Filter funnel,* of approximately 100 mm diameter.

3.2.17 *Filter papers,* of a diameter appropriate to the size of the funnel : medium grade (e.g. Whatman No. 40) and fine grade (e.g. Whatman No. 42).

3.2.18 *Blue litmus paper.*

3.3 Reagents

3.3.1 *General.* All reagents shall be of recognized analytical reagent quality

NOTE Where accurately standardized solutions are required it may be more convenient to obtain them already standardized in concentrated form and to dilute them as necessary in accordance with the manufacturer's instructions.

3.3.2 *Potassium dichromate solution,* ($c(K_2Cr_2O_7)$ = 0.167 mol/L). Dissolve 49.035 g of potassium dichromate in distilled water to make 1 L of solution.

3.3.3 *Ferrous sulphate solution.* Dissolve approximately 140 g of ferrous sulphate in sulphuric acid solution ($c(H_2SO_4)$ = 0.25 mol/L) to make 1 L of solution. Add 14 mL of concentrated sulphuric acid to distilled water to make 1 L of sulphuric acid solution ($c(H_2SO_4)$ = 0.25 mol/L). Record the date the solution is made on the bottle.

This solution is unstable in air. Keep it tightly stoppered and standardize against the potassium dichromate solution at least once a week.

CAUTION. Take great care when adding the acid to the water. Do not add water to concentrated sulphuric acid, that procedure is dangerous.

3.3.4 *Sulphuric acid,* concentrated. Density 1.84 g/mL.

3.3.5 *Orthophosphoric acid,* 85 % (*V/V*) solution. Density 1.70 to 1.75 g/mL.

3.3.6 *Indicator solution.* Dissolve 0.25 g of sodium diphenylaminesulphonate in 100 mL of distilled water.

3.3.7 *Hydrochloric acid,* 25 % (*V/V*) solution. Dilute 250 mL of concentrated hydrochloric acid (density 1.18 g/mL) to 1 L with distilled water.

3.3.8 *Lead acetate paper.* Filter paper that has been dipped in a 10 % solution of lead acetate.

3.3.9 *Sulphuric acid* ($c(H_2SO_4)$ = approximately 1 mol/L). Add 53 ml of concentrated sulphuric acid to about 500 ml of distilled water, then make up to 1 L with distilled water.

3.4 Procedure

3.4.1 ***Standardization of ferrous sulphate***

3.4.1.1 Run 10 mL of the potassium dichromate solution (**3.3.2**) from a burette into a 500 mL conical flask.

3.4.1.2 Add 20 mL of concentrated sulphuric acid very carefully, swirl the mixture and allow to cool for some minutes.

3.4.1.3 Add 200 mL of distilled water very carefully to the mixture followed by 10 mL of phosphoric acid and 1 mL of the indicator, and shake the mixture thoroughly.

3.4.1.4 Add ferrous sulphate solution from the second burette in 0.5 mL increments, and swirl the contents of the flask until the colour of the solution changes from blue to green.

3.4.1.5 Add a further 0.5 mL of potassium dichromate solution, changing the colour back to blue.

3.4.1.6 Slowly add ferrous sulphate solution drop by drop with continued swirling until the colour of the solution changes from blue to green after the addition of a single drop.

3.4.1.7 Record the total volume of ferrous sulphate solution used, x, to the nearest 0.05 mL.

3.4.2 ***Preparation of test specimen***

3.4.2.1 Prepare each test specimen for analysis from the laboratory sample as given in **3.4.2.2** to **3.4.2.11**.

3.4.2.2 Obtain an initial sample as described in **7.3**, and of the approximate size as specified in **7.5,** of BS 1377-1:1990.

3.4.2.3 Dry this sample in the oven to constant mass at 50 ± 2.5 °C, and cool to room temperature in the desiccator.

3.4.2.4 Weigh the sample to the nearest 0.1 % and record the mass m_1 (in g).

3.4.2.5 Sieve the sample on a 2 mm test sieve (if appropriate, guarded by a test sieve of larger aperture) and crush retained particles other than stones to pass the 2 mm sieve.

NOTE It is assumed that any material retained on the 2 mm test sieve will not contain organic matter. If this is seen not to be true, the pieces of organic matter should be removed by hand, crushed to pass a 2 mm test sieve and incorporated in the fraction passing the sieve.

3.4.2.6 Reject the stones, ensuring that no fine material adheres to them, e.g. by brushing. Record the mass of sample passing the 2 mm test sieve (in g) to the nearest 0.1 % (m_2). Throughout these and subsequent operations take care to ensure that there is no loss of fines.

3.4.2.7 Divide the material passing the 2 mm sieve by successive riffling through the 15 mm divider to produce a sample weighing approximately 100 g.

3.4.2.8 Pulverize this sample so that it passes the 425 μm test sieve.

3.4.2.9 Subdivide the sample by riffling on the 7 mm divider to obtain the following test specimens, as appropriate:

a) If sulphides and chlorides are known to be absent, two test specimens each weighing about 5 g;

b) for a check test to determine whether sulphides are present, a specimen of about 5 g;

c) for a check test to determine whether chlorides are present, a speciment of about 50 g;

d) if sulphides and/or chlorides are present, a specimen of about 50 g.

Throughout this and any subsequent operation mix the material available thoroughly before any division and take care to avoid segregation during riffling.

3.4.2.10 Place each specimen in a glass weighing bottle and dry in the oven at a temperature of 50 ± 2.5 °C. The specimens are deemed to be dry when the differences in successive weighings, carried out at intervals of 4 h, do not exceed 0.1 % of the original mass of the sample.

3.4.2.11 Allow the specimens to cool to room temperature in the desiccator and weigh each bottle and contents to 0.001 g.

3.4.3 *Qualitative check for sulphides*

3.4.3.1 Verify the presence of sulphides in the soil as described in **3.4.3.2** to **3.4.3.5**.

3.4.3.2 Place the 5 g check sample (which need not be weighed) in a boiling tube and add about 20 mL of hydrochloric acid, 25 % solution.

3.4.3.3 Boil the acid and check for hydrogen sulphide by holding in the vapour a piece of filter paper that has been dipped in a 10 % solution of lead acetate. This will turn black if hydrogen sulphide is present.

3.4.3.4 If the presence of sulphides is indicated, remove them from the test specimen as described in **3.4.4** before proceeding with the analysis for organic matter, otherwise a result that is too high will be obtained.

3.4.3.5 If the presence of sulphides is not indicated, omit the procedure of **3.4.4**.

3.4.4 *Elimination of sulphides*

3.4.4.1 Weigh about 50 g of the soil, after cooling as described in **3.4.2.11**, to 0.01 g and place it in a 500 mL conical flask.

3.4.4.2 Add sulphuric acid solution ($c(H_2SO_4) = 1.0$ mol/L) until no further evolution of hydrogen sulphide occurs, as determined by testing with lead acetate paper.

3.4.4.3 Filter the contents of the conical flask on a medium grade filter paper, taking care to retain all solid particles. Wash several times with hot distilled water until the washings do not indicate acidity when tested with blue litmus.

3.4.4.4 Dry the soil retained on the filter paper to constant mass at a temperature of 50 ± 2.5 °C and cool in the desiccator.

3.4.4.5 Carefully remove the soil from the filter paper and determine its mass to 0.01 g.

3.4.4.6 Subdivide the treated sample, as described in item a) of **3.4.2.9**, and dry and cool each test specimen as described in **3.4.2.10** and **3.4.2.11**.

3.4.5 *Qualitative check for chlorides*. Verify the presence of chlorides in the soil by following the procedure described in **7.2.3.3**.

If the presence of chlorides is indicated, remove them from the test specimen as described in **3.4.6** before proceeding with the analysis for organic matter, otherwise a result that is too high will be obtained.

NOTE 1 Alternatively, the effect of chlorides on the organic matter determination can be partly eliminated by using concentrated sulphuric acid in which silver sulphate has been dissolved in place of the concentrated sulphuric acid specified in **3.3.4**. If the ratio of carbon to chloride does not exceed unity, 25 g of silver sulphate per litre of sulphuric acid will be sufficient to precipitate the chloride.

NOTE 2 If the presence of both sulphides and chlorides is indicated, the procedures described in **3.4.4** and **3.4.6** should both be carried out on the sample of soil used for determination of the organic content.

If the presence of chlorides is not indicated, the procedure of **3.4.6** may be omitted.

3.4.6 *Elimination of chlorides*

3.4.6.1 Weigh about 50 g of the soil, after cooling as described in **3.4.2.11**, to 0.01 g.

3.4.6.2 Place the soil on a medium-grade filter paper, e.g. Whatman No. 40, in a funnel and wash with distilled water.

3.4.6.3 Continue washing until no turbidity is observed when a drop of the wash water is tested with silver nitrate solution.

3.4.6.4 Dry the soil retained on the filter paper to constant mass at a temperature of 50 ± 2.5 °C and cool in the desiccator.

3.4.6.5 Carefully remove all the soil from the filter paper and determine its mass to 0.01 g.

3.4.6.6 Subdivide the treated sample, as described in item a) of **3.4.2.9**, and dry and cool each test specimen as described in **3.4.2.10** and **3.4.2.11**.

3.4.7 *Analysis for organic matter*

3.4.7.1 Weigh each weighing bottle containing soil, obtained as described in **3.4.2**, **3.4.4** or **3.4.6**, to 0.001 g.

3.4.7.2 Transfer a small quantity, from 5.0 g to 0.2 g, depending on the organic content (see the note), to a dry 500 mL conical flask, reweigh the weighing bottle and calculate the mass of soil removed (m_3) by difference.

NOTE The size of the sample for chemical analysis will vary with the amount of organic matter present in the soil. As much as 5 g may be required for soil low in organic matter, and as little as 0.2 g with a very peaty soil. After a number of determinations have been made experience will indicate the most suitable size of sample to be taken. Where this is not so, it is suggested that a series of samples of varying sizes should be tested. The determination giving a total of 5 mL to 8 mL of potassium dichromate solution reduced should be taken as the one giving the correct result.

3.4.7.3 Run 10 mL of the potassium dichromate solution (**3.3.2**) into the conical flask from a burette, and add 20 mL concentrated sulphuric acid very carefully from a measuring cylinder. Swirl the mixture thoroughly for about 1 min, and then allow to stand on a heat-insulating surface for 30 min to allow oxidation of the organic matter to proceed. During this period protect the flask from cold air and draughts.

3.4.7.4 Add 200 mL of distilled water very carefully to the mixture, followed by 10 mL of orthophosphoric acid and 1 mL of indicator, and shake the mixture thoroughly. If the indicator is absorbed by the soil add a further 1 mL of the solution.

3.4.7.5 Add ferrous sulphate solution from the second burette in 0.5 mL increments and swirl the contents of the flask until the colour of the solution changes from blue to green.

3.4.7.6 Add a further 0.5 mL of potassium dichromate solution, changing the colour back to blue.

3.4.7.7 Add ferrous sulphate solution slowly drop by drop with continued swirling until the colour of the solution changes from blue to green after the addition of a single drop.

3.4.7.8 Record the total volume of ferrous sulphate solution used, y, to the nearest 0.05 mL.

3.5 Calculations

NOTE See form 3(a) of Appendix A.

3.5.1 The total volume V (in mL) of potassium dichromate solution used to oxidize the organic matter in the soil sample is calculated from the equation:

$$V = 10.5\ (1 - y/x)$$

where

y is the total volume of ferrous sulphate solution used (see **3.4.7.8**) (in mL);

x is the total volume of ferrous sulphate solution used in the standardization test (see **3.4.1**) (in mL).

3.5.2 Calculate the percentage of the original soil sample passing the 2 mm test sieve from the equation:

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100$$

where

m_1 is the initial dry mass of sample (in g);

m_2 is the mass of the sample passing the 2 mm test sieve (in g).

3.5.3 Calculate the percentage of organic matter present in the fraction of the soil specimen finer than 2 mm for each determination from the equation (see note):

$$\text{percentage organic matter content} = \frac{0.67\ V}{m_3}$$

where

m_3 is the mass of soil used in the test (in g).

NOTE The method is based on wet oxidation of the organic content of the soil, and assumes that soil organic matter contains an average of 58 % (m/m) of carbon. The method employed oxidizes approximately 77 % of the carbon in the organic matter, and these factors are included in the equation given in **3.5.3**. The factors will give correct results only for soil containing natural organic matter.

3.5.4 If duplicate specimens have been tested, and if the individual results expressed as a percentage of organic matter differ by no more than 2 %, calculate the mean result. If they differ by more than 2 %, repeat the test starting with two new representative portions of soil.

3.6 Test report

The test report shall state that the test was carried out in accordance with clause **3** of BS 1377-3:1990, and shall contain the following information:

a) the method of test used;

b) the average organic matter content present in the soil fraction passing a 2 mm test sieve to the nearest 0.1 % of the original oven dry mass of soil;

c) the percentage by dry mass of the original sample passing the 2 mm test sieve to the nearest 1 %;

d) if sulphides or chlorides have been identified in the soil, this shall be stated;

e) the information required by **9.1** of BS 1377-1:1990.

4 Determination of the mass loss on ignition

4.1 General

This clause describes the procedure for determining the proportion by mass that is lost from a soil by ignition at a specified temperature.

NOTE Good practice in chemical testing requires that duplicate specimens should be tested. In each of the test methods the measurement of only one value of the overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a mean value and an indication of the reliability of the results. Guidance on the number of measurements required and the treatment of the results obtained is beyond the scope of this Part of BS 1377.

The mass loss on ignition is related to the organic content of certain soils, such as sandy soils that contain little or no clay, or chalky material, peats and organic clays containing more than about 10 % organic matter. However it should be recognized that, in some soils, factors unrelated to organic content could be responsible for the major proportion of the mass loss on ignition.

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

4.2 Apparatus

4.2.1 *Drying oven* capable of maintaining a temperature of 50 ± 2.5 °C.

4.2.2 *Balance,* readable to 1 g.

4.2.3 *Balance,* readable to 0.001 g.

4.2.4 *Desiccator,* containing anhydrous silica gel.

4.2.5 *Test sieves,* 2 mm and 425 µm aperture sizes, with receiver.

4.2.6 *Pestle and mortar*, or a suitable mechanical crusher.

4.2.7 *Sample dividers of the multiple-slot type (riffle boxes),* having widths of opening of 7 mm and 15 mm.

4.2.8 *Crucible or similar container*, of about 30 mL capacity.

4.2.9 *Electric muffle furnace*, capable of maintaining a temperature of 440 ± 25 °C.

4.3 Procedure

4.3.1 *Preparation of crucible*

4.3.1.1 Before starting each series of tests, carry out a test on the empty crucible or container as described in **4.3.1.2** to **4.3.1.4**.

4.3.1.2 Place the crucible in the muffle furnace, heat to 440 ± 25 °C and maintain the temperature for 1 h.

4.3.1.3 Remove the crucible from the furnace and allow it to cool to room temperature in the desiccator.

4.3.1.4 Weigh the crucible to the nearest 0.001 g (m_c).

4.3.2 *Preparation of test specimen*

4.3.2.1 Prepare each test specimen for analysis from the laboratory sample as described in **4.3.2.2** to **4.3.2.8**.

4.3.2.2 Obtain an initial sample as described in **7.3**, and of the approximate size specified in **7.5,** of BS 1377-1:1990.

4.3.2.3 Dry this sample in the oven at 50 ± 2.5 °C, and cool to room temperature in the desiccator.

4.3.2.4 Weigh the sample to the nearest 0.1 % and record the mass m_1 (in g).

4.3.2.5 Sieve the sample on a 2 mm test sieve (if appropriate, guarded by a test sieve of larger aperture), and crush retained particles other than stones to pass the 2 mm test sieve.

4.3.2.6 Reject the stones ensuring that no fine material adheres to them, e.g. by brushing. Record (in g) the mass of the sample passing the 2 mm test sieve to the nearest 0.1 % (m_2). Throughout these and subsequent operations ensure that there is no loss of fines.

4.3.2.7 Divide the material passing the 2 mm sieve by successive riffling through the 15 mm divider to produce a sample weighing at least 10 g.

4.3.2.8 Pulverize this sample so that it passes the 425 μm test sieve.

4.3.2.9 Subdivide the sample by riffling on the 7 mm divider to obtain test specimens each weighing approximately 5 g. Throughout this and any subsequent operation mix the material available thoroughly before any division and avoid segregation during riffling.

4.3.2.10 Place each specimen in a prepared crucible and dry in the oven at a temperature of 50 ± 2.5 °C. The specimens are deemed to be dry when the differences in successive weighings, carried out at intervals of 4 h, do not exceed 0.1 % of the original mass of the sample.

4.3.2.11 Allow the specimens to cool to room temperature in the desiccator and weigh each crucible to 0.001 g (m_3).

4.3.3 *Ignition of soil*

4.3.3.1 Ignite each test specimen as described in **4.3.3.2** to **4.3.3.4**.

4.3.3.2 Place the crucible with soil in the unheated muffle furnace, heat to 440 ± 25 °C, and maintain this temperature for a period of not less than 3 h, or until constant mass is achieved.

NOTE The period required for ignition will vary with the type of soil and size of sample.

4.3.3.3 Remove the crucible and contents from the furnace and allow to cool to room temperature in the desiccator.

4.3.3.4 Weigh the crucible and contents to the nearest 0.001 g (m_4).

4.4 Calculations

NOTE See form 3(b) of Appendix A.

4.4.1 Calculate the percentage of the original soil sample passing the 2 mm test sieve from the equation:

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100$$

where

m_1 is the original dry mass of sample (in g);

m_2 is the mass of sample passing the 2 mm sieve (in g).

4.4.2 Calculate the mass loss on ignition, LOI, as a percentage of the dry mass of soil passing a 2 mm test sieve from the equation:

$$\text{LOI} = \frac{m_3 - m_4}{m_3 - m_c} \times 100$$

where

m_3 is the mass of the crucible and oven-dry soil specimen (in g);

m_4 is the mass of the crucible and specimen after ignition (in g);

m_c is the mass of the crucible (in g).

4.5 Test report

The test report shall state that the test was carried out in accordance with clause **4** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the mass loss on ignition as a percentage of the soil fraction passing the 2 mm test sieve, to two significant figures;

c) the percentage by dry mass of the original sample passing the 2 mm test sieve, to the nearest 1 %;

d) the information required by **9.1** of BS 1377-1:1990.

5 Determination of the sulphate content of soil and ground water

5.1 General

5.1.1 *Principle.* This clause describes procedures for determining the sulphate content of soil and ground water. The results obtained give the sulphate content at the time of sampling only.

Acid-soluble sulphates include all naturally occurring sulphates with rare exceptions. Calcium sulphate, the sulphate salt that is most commonly found, has a low solubility in water.

5.1.2 *Types of test.* Procedures are described for the determination of the following:

NOTE Good practice in chemical testing requires that duplicate specimens should be tested. In each of the test methods the measurement of only one value of the overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a mean value and an indication of the reliability of the results. Guidance on the number of measurements required and the treatment of the results obtained is beyond the scope of this Part of this standard.

a) the acid-soluble sulphate content of soil (also referred to as the total sulphate content) for which an acid extract is first prepared (see **5.2**);

b) the water-soluble sulphate content of soil, for which a water extract is first prepared (see **5.3**);

NOTE If the sulphate present in the soil is predominantly the calcium salt, the total sulphate content as determined in **5.2** is likely to give a misleading and pessimistic impression of the danger to concrete or cement-stabilized material arising from the presence of sulphates. In cases where the total sulphate exceeds 0.5 % it is suggested that the water soluble sulphate content of a 2 to 1 water-soil extract should be determined. If calcium sulphate is the only sulphate salt present its low solubility will ensure that the sulphate content of the aqueous extract does not exceed 1.2 g/L. Sulphate contents in excess of this figure in the soil-water extract or in the ground water as determined in this test therefore indicate the presence of other and more harmful sulphate salts.

c) the dissolved sulphates in ground water (see **5.4**).

Soils containing sulphides may require special attention.

NOTE Soils containing sulphides. The acid extraction method determines the total sulphate content of soil at the time of test and any sulphides which may be present are destroyed in the extraction process (see note to **5.2.4.4**). If sulphides are present it is possible that these may oxidize in the long term to produce additional sulphates. If the presence of sulphides is suspected and the possible oxidation of these in the future is a cause of concern (as for example in the case of reinforced earth) the total sulphur content should be measured by the method given in BS 1047. The difference in the total sulphur content and the total, i.e. acid-soluble, sulphate content both expressed in terms of percentage SO_3 gives a measure of the amount of sulphide present and of any potential long-term risk.

5.1.3 *Methods of analysis.* Two methods of analysis are described:

a) the gravimetric method for acid extracts, water extracts and ground water samples (see **5.5**);

b) the ion-exchange procedure for water extracts and ground water samples only (see **5.6**).

In the gravimetric method, barium chloride solution is added to the acid or water extract and the precipitate of barium sulphate is collected, dried and weighed. The sulphate content is then calculated from the mass of the material used in the analysis and the mass of barium sulphate precipitated.

In the ion-exchange procedure the water extract or the sample of ground water is passed through a resin ion-exchange column and the quantity of anions present is measured by titration against a standardized solution of sodium hydroxide. This method cannot be used if the soil or ground water contains anions of other strong acids, such as chlorides, nitrates and phosphates.

NOTE The presence or otherwise of interfering anions should be investigated before selecting the appropriate method. If the presence of other anions precludes the use of the ion-exchange method the sulphate content of the soil-water extract and of the ground water can be determined gravimetrically as described in **5.5**.

5.1.4 *Alternative combinations of procedures.* There is some flexibility in the selection of method of analysis on a given type of sulphates extract. An acid extract from a soil sample is analysed by the gravimetric method, but a water extract may be analysed either by the gravimetric method or by the ion-exchange method. A sample of ground water may be analysed by either method.

5.2 Preparation of soil and its acid extract

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

5.2.1 ***Apparatus***

5.2.1.1 *Apparatus for preparation of test specimen*

5.2.1.1.1 *Drying oven,* capable of being controlled to maintain a temperature range of 75 °C to 80 °C, as well as the range 105 °C to 110 °C.

5.2.1.1.2 *Balance,* readable to 1 g.

5.2.1.1.3 *Balance,* readable to 0.001 g.

5.2.1.1.4 *Desiccator,* containing anhydrous silica gel.

5.2.1.1.5 *Test sieves,* 2 mm and 425 µm aperture sizes, with receiver.

5.2.1.1.6 *Pestle and mortar,* or a suitable mechanical crusher.

5.2.1.1.7 *Sample dividers of multiple-slot type (riffle boxes),* having widths of opening of 7 mm and 15 mm.

5.2.1.1.8 *Glass weighing-bottle,* approximately 50 mm in diameter, 25 mm high and fitted with a ground-glass stopper.

5.2.1.1.9 *Red litmus paper.*

5.2.1.2 *Apparatus for preparing acid-soluble sulphate extract.*

5.2.1.2.1 *Two* 500 mL *conical beakers with glass rods and cover glasses.*

5.2.1.2.2 *Electric hot-plate,* capable of being controlled to boil the contents of the beakers.

5.2.1.2.3 *Buchner funnel,* of about 100 mm diameter.

5.2.1.2.4 *Vacuum filtration flask,* of about 500 mL capacity, to take the funnels.

5.2.1.2.5 *Filter papers,* of medium grade (e.g. Whatman No. 40), hardened medium grade (e.g. Whatman No. 540) and fine grade (e.g. Whatman No. 42) to fit the funnels.

5.2.1.2.6 *Source of vacuum,* e.g. a filter pump.

5.2.1.2.7 *Length of rubber vacuum tubing,* to fit the vacuum pump and filter flask.

5.2.1.2.8 *Two glass rods,* about 150 mm to 200 mm long and 3 mm to 5 mm in diameter.

5.2.1.2.9 *Wash bottle,* preferably made of plastics, containing distilled water.

5.2.1.2.10 *Glass filter funnel,* about 100 mm diameter.

5.2.1.2.11 *Dropping pipette,* 10 mL capacity.

5.2.1.2.12 *Amber-coloured glass container.*

5.2.2 ***Reagents***

5.2.2.1 *General.* All reagents shall be of recognized analytical reagent quality.

NOTE Where accurately standardized solutions are required it may be more convenient to obtain them already standardized in concentrated form and to dilute them as necessary in accordance with the manufacturer's instructions.

5.2.2.2 *Hydrochloric acid,* 10 % (*v*/*v*) solution. Dilute 100 mL of concentrated hydrochloric acid (relative density 1.18) to 1 L with distilled water.

5.2.2.3 *Dilute ammonia solution.* Dilute 500 mL of ammonia (relative density 0.880) to 1 L with distilled water.

5.2.2.4 *Silver nitrate.* 0.5 % (*m*/*V*) solution. Dissolve 0.5 g of silver nitrate in 100 mL of distilled water. Store the solution in an amber-coloured glass container.

5.2.2.5 *Concentrated nitric acid* density (1.42 g/mL).

5.2.2.6 *Distilled water,* of a purity complying with **5.1** of BS 1377-1:1990.

5.2.3 ***Preparation of test specimen***

5.2.3.1 Prepare each test specimen for analysis from the laboratory sample as given in **5.2.3.2** to **5.2.3.11**.

5.2.3.2 Obtain an initial sample as described in **7.3**, and of the approximate size specified in **7.5**, of BS 1377-1:1990.

5.2.3.3 Dry this sample in the oven at a temperature between 75 °C and 80 °C and allow to cool to room temperature in the desiccator.

NOTE Soils containing sulphates in the form of gypsum lose water of crystallization if heated above the specified temperature.

5.2.3.4 Weigh the sample to the nearest 0.1 % and record the mass m_1(in g).

5.2.3.5 Sieve the sample on a 2 mm test sieve (if appropriate, guarded by a test sieve of larger aperture), and crush particles other than stones to pass through the sieve.

NOTE It is assumed that any material retained on the 2 mm test sieve will not contain sulphates. This is generally true but certain soils may contain lumps of gypsum larger than 2 mm in diameter and in such cases the gypsum should be removed by hand, crushed to pass a 2 mm test sieve and incorporated in the fraction passing the sieve.

5.2.3.6 Reject the stones ensuring that no fine material adheres to them, e.g. by brushing. Record (in g) the mass of the sample passing the 2 mm test sieve to the nearest 0.1 % (m_2). Throughout these and subsequent operations ensure that there is no loss of fines.

5.2.3.7 Divide the material passing the 2 mm sieve by successive riffling through the 15 mm divider to produce a sample weighing approximately 100 g.

5.2.3.8 Pulverize this sample so that it passes the 425 µm test sieve. Throughout this and any subsequent operation, mix the material thoroughly before any division and take care to avoid segregation during riffling.

5.2.3.9 Subdivide the sample by successive riffling through the 7 mm divider to obtain test specimens each weighing approximately 10 g.

5.2.3.10 Place each specimen in a glass weighing bottle and dry in the oven at a temperature of 75 °C. The specimens are deemed to be dry when the differences in successive weighings at intervals of 4 h do not exceed 0.1 % of the specimen mass.

5.2.3.11 Allow the specimens to cool to room temperature in the desiccator.

5.2.4 *Preparation of acid extract*

5.2.4.1 Obtain the acid extract for the determination of the total sulphate content from each of the prepared specimens as given in **5.2.4.2** to **5.2.4.5**.

5.2.4.2 Weigh the weighing bottle containing the dried test specimen to the nearest 0.001 g.

5.2.4.3 Transfer a representative sample of about 2 g in mass (see note) to a 500 mL beaker, reweigh the weighing bottle and calculate the mass (m_3) of the sample by difference.

NOTE The mass of sample to use depends on the amount of sulphate present. Ideally a mass should be chosen that will produce a precipitate of barium sulphate weighing approximately 0.2 g.

5.2.4.4 Add 100 mL of 10 % hydrochloric acid to the sample, taking care if frothing occurs to ensure that no material is lost.

NOTE Materials containing sulphides will release H_2S on acidification and this can be detected by its smell. In these cases there is a danger that this procedure will overestimate the sulphate content because of sulphide oxidation. If the material contains sulphides, place 100 mL of the acid in a 500 mL beaker and heat to boiling. Remove from the heat source and whilst stirring the acid solution sprinkle the weighed analytical sample on to the acid.

5.2.4.5 Cover the beaker with a cover glass, boil, and simmer gently for 15 min in a fume cupboard. Rinse the underside of the cover glass back into the beaker.

5.2.4.6 Filter the solution (see NOTE) through a hardened filter paper into a 500 mL conical beaker. Wash the beaker and residue thoroughly with distilled water until the washings are free from chloride, as indicated by an absence of turbidity when a drop is added to a small volume of silver nitrate solution. Collect the washings with the filtrate and analyse the filtrate as described in **5.5.3**.

NOTE For soils that contain sesquioxides in appreciable quantities, e.g. certain tropical residual soils, the sesquioxides should be precipitated before proceeding with the sulphate analysis, as follows.

a) Add a few drops of nitric acid while the suspension continues to boil.

b) Add ammonia solution slowly (preferably from a burette), with constant stirring, to the boiling solution until the sesquioxides are precipitated and red litmus is turned blue by the liquid. Filter and proceed as described in **5.2.4.6**.

c) If a voluminous precipitate of sesquioxides forms when ammonia is added to neutralize the acid, some sulphate may be entrapped which will not be removed by washing and could lead to low results. In this case a second precipitation is recommended. This is done by carefully removing the filter paper with the precipitate and replacing it in the original beaker. A 10 % solution of hydrochloric acid is added and the contents stirred until the sesquioxides have gone into the solution (20 mL of 10 % hydrochloric acid should be sufficient). Bring the contents to the boil and repeat step b) above.

5.3 Preparation of soil and its water extract

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

5.3.1 *Apparatus*

5.3.1.1 *Apparatus for preparation of test specimen.* The apparatus required for the preparation of each test specimen is the same as listed in **5.2.1.1**.

5.3.1.2 *Apparatus for preparing water extract.*

5.3.1.2.1 *General.* The apparatus listed in **5.3.1.2.2** to **5.3.1.2.5** is required in addition to the items listed in **5.2.1.2**.

5.3.1.2.2 *Mechanical shaker or stirrer,* capable of keeping 50 g of soil in continuous suspension in 100 mL of water.

5.3.1.2.3 *Extraction bottle,* of approximately 250 mL capacity.

5.3.1.2.4 75 mm *diameter watch glass.*

5.3.1.2.5 50 mL *and a* 25 mL *pipette.*

5.3.2 *Preparation of test specimen*

5.3.2.1 Prepare each test specimen for analysis from the soil sample as described in **5.3.2.2** to **5.3.2.6**.

5.3.2.2 Prepare a sample of material passing the 2 mm test sieve, as described in **5.2.3.2** to **5.2.3.6**.

5.3.2.3 Divide the material passing the 2 mm sieve by successive riffling through the 15 mm divider to produce a sample weighing approximately 60 g.

5.3.2.4 Pulverize this sample so that it passes the 425 µm test sieve. Throughout this and any subsequent operation, mix the material thoroughly before any division and take care to avoid segregation during riffling.

5.3.2.5 Place the specimen in a glass weighing bottle and dry at a temperature of 75 °C to 80 °C. The specimen is deemed to be dry when the differences in successive weighings at intervals of 4 h do not exceed 0.1 % of the specimen mass.

5.3.2.6 Allow the specimen to cool to room temperature in the desiccator.

5.3.3 *Preparation of water extract*

5.3.3.1 Obtain the 2 : 1 water-soil extract for the determination of the water-soluble sulphate content from each of the prepared specimens as given in **5.3.3.2** to **5.3.3.6**.

5.3.3.2 Weigh a sample of 50.00 g on a watch glass and transfer it to a clean and dry extraction bottle.

5.3.3.3 Add exactly 100 mL of distilled water to the extraction bottle, stopper it tightly and place it in the shaker and agitate for 16 h.

5.3.3.4 Filter the soil suspension into a clean and dry flask through a suitable filter paper, e.g. Whatman No. 50, on the Buchner funnel.

NOTE If a centrifuge is available it will probably prove more convenient to use a centrifuge tube for the extraction. The suspension can then be centrifuged instead of filtered and 25 mL of the clear supernatant liquid is used for the analysis.

5.3.3.5 If the ion-exchange method of analysis is to be used (see **5.6**) it is essential that no additional water is added at any stage and the soil remaining on the filter paper is not washed. Transfer 50 mL of the extract to a clean day 250 mL conical beaker and add distilled water to make up to 100 mL.

5.3.3.6 If the gravimetric method of analysis is to be used, transfer 50 mL of filtered extract to a 500 mL conical beaker. Add distilled water to about 300 mL and proceed to **5.5**.

5.4 Preparation of ground water for testing

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

5.4.1 *Apparatus*

5.4.1.1 *Filtration funnel and stand.*

5.4.1.2 *Filter papers* of a diameter to suit the funnel, Whatman No. 44 grade or similar.

5.4.1.3 *Three* 500 mL *glass conical beakers.*

5.4.1.4 *Two* 250 mL *conical beakers.*

5.4.1.5 100 mL *glass measuring cylinder.*

5.4.1.6 50 mL *pipette.*

5.4.1.7 *Wash bottle,* preferably made of plastics, containing distilled water.

5.4.2 *Procedure*

5.4.2.1 From a sample of at least 500 mL of ground water prepare two test samples as given in **5.4.2.2** to **5.4.2.4**.

5.4.2.2 Filter the water through a suitable filter paper such as Whatman No. 44 into a clean flask, to remove any particles in suspension.

5.4.2.3 If the gravimetric method of analysis (see **5.5**) is to be used, transfer 50 mL of the filtered water to each of two clean, dry 500 mL conical beakers, and add approximately 100 mL of distilled water to each.

5.4.2.4 If the ion-exchange method of analysis (see **5.6**) is to be used, transfer exactly 100 mL of the filtered water to each of two clean dry 250 mL conical beakers.

5.5 Gravimetric method for analysis of acid or water extract or ground water

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

5.5.1 *Apparatus*

5.5.1.1 The apparatus listed in **5.5.1.2** to **5.5.1.4** is required in addition to the items listed in **5.2.1** or **5.3.1** or **5.4.1**.

5.5.1.2 Either:

a) *a sintered silica filtering crucible* of porosity grade No. 4 and about 35 mm diameter and 40 mm high; or

b) *an ignition crucible* about 35 mm diameter and 40 mm high, capable of maintaining a constant mass when heated to 800 ± 50 °C.

5.5.1.3 *A suitable means of igniting the precipitate,* preferably an electric muffle furnace capable of reaching and maintaining 800 ± 50 °C. Alternatively a Bunsen burner or similar burner, tripod and suitable pipeclay triangle may be used.

5.5.1.4 *Balance,* readable to 0.001 g.

5.5.1.5 *Blue litmus paper.*

5.5.2 *Reagents*

5.5.2.1 *General.* The reagent listed in **5.5.2.2** is required, in addition to those listed in **5.2.2**. All reagents shall be of recognized analytical reagent quality.

NOTE Where accurately standardized solutions are required it may be more convenient to obtain them already standardized in concentrated form and to dilute them as necessary in accordance with the manufacturer's instructions.

5.5.2.2 *Barium chloride,* 5 % (*m/V*) solution. Dissolve 50 g of barium chloride in 1 L of water. Filter before use if necessary.

5.5.3 *Test procedure*

5.5.3.1 Analyse each filtrate sample obtained from the acid extract procedure described in **5.2.4**, or from the water extract procedure described in **5.3.3** (see **5.3.3.6**), or from the ground water sample prepared as described in **5.4.2** (see **5.4.2.4**), as described in **5.5.3.2** to **5.5.3.8**.

5.5.3.2 Test the solution with litmus paper and if necessary make slightly acid by the addition of 20 drops of hydrochoric acid.

5.5.3.3 Dilute to 300 mL (if necessary), bring the solution to the boil and add 10 mL barium chloride solution drop by drop with constant stirring. Continue boiling gently until the precipitate is properly formed.

5.5.3.4 Allow the solution to stand at just below boiling point for at least 30 min, then leave to cool to room temperature.

5.5.3.5 Transfer the liquid and precipitate of barium sulphate with extreme care to a previously ignited and weighed sintered silica filter crucible using suction. Alternatively transfer the precipitate with extreme care to a suitable filter paper, e.g. Whatman No. 42, in the glass filter funnel and filter. In either case wash the precipitate several times with hot distilled water until the washings are free from chloride as indicated by absence of turbidity when a drop is tested with the solution of silver nitrate.

5.5.3.6 If a sintered silica filter crucible is used remove it from the filter flask and dry at 105 °C to 110 °C for approximately 30 min and gradually raise the temperature to 800 °C, either in an electric muffle furnace or by other suitable means until no further loss in mass occurs.

5.5.3.7 If the precipitate is filtered through a filter paper, transfer the filter paper and precipitate to a previously ignited and weighed crucible. If an electric muffle furnace is used, place the crucible and contents in it at room temperature and gradually raise the temperature to red heat. If a Bunsen or other burner is used first dry the filter paper and precipitate slowly over a small flame, taking care to char the filter paper slowly rather than to allow it to inflame, otherwise some of the precipitate may be lost. Ignition for about 15 min at red heat is usually sufficient in either case.

5.5.3.8 Following **5.5.3.6** or **5.5.3.7** cool the crucible to room temperature in the desiccator and weigh it to the nearest 0.001 g. Calculate the mass of the precipitate (m_4) from the increase in the recorded mass of the crucible.

5.5.4 *Calculations*

NOTE 1 Traditionally sulphate contents are expressed in terms of SO_3 and this is the method that has therefore been adopted here. If it is desired to express sulphates in terms of SO_4 the results, in terms of SO_3, in the relevant equations given in this clause should be multiplied by 1.2.

NOTE 2 See form 3 (c) of Annex A.

5.5.4.1 *Soil samples*

5.5.4.1.1 Calculate the percentage of the original soil sample passing the 2 mm test sieve from the equation:

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100$$

where

m_2 is the mass of the fraction passing the 2 mm sieve (in g);

m_1 is the initial dry mass of sample (in g).

5.5.4.1.2 For the acid extract procedure, calculate the percentage of total sulphates (as SO_3) in the fraction of the soil sample finer than 2 mm for each determination from the equation:

$$SO_3 = \frac{34.3\ m_4}{m_3}\ (\%)$$

where

m_3 is the mass of each test specimen used (in g);

m_4 is the mass of ignited precipitate (in g).

If more than one specimen has been tested and the individual results differ by no more than 0.2 % (SO_3), calculate the mean result. If they differ by more than 0.2 %, repeat the test starting with two new analytical portions of the soil.

5.5.4.1.3 For the water extract procedure, the sulphate content can be expressed either in terms of the concentration of sulphate in the extract (as SO_3) in g/L as in Method 1 below, or in terms of percentatge (as SO_3) of water-soluble sulphate in the soil sample as in Method 2 below

Method 1. Calculate the sulphate content in g/L of the 2 : 1 water/soil extract from the equation: Concentration of sulphate (as SO_3) = $6.86 m_4$ g/L Where m_4 is the mass of ignited precipitate (in g).

Method 2. Calculate the sulphate content as a percentage of the soil from the equation: Water-soluble sulphate content (as SO_3) = $1.372 m_4$ (%) Where m_4 is the mass of ignited precipitate (in g).

If individual results differ by no more than 0.2 % or 0.2 g/L (SO_3), calculate the mean result. If they differ more than 0.2 % or 0.2 g/L, repeat the test starting with two new analytical portions of the soil.

5.5.4.2 *Ground water samples.* For each determination calculate the sulphate content (as SO_3) of the ground water (in g/L) from the equation:

$$SO_3 = 6.86m_4$$

If individual results differ by no more than 0.2 g/L (SO_3), calculate the mean result. If they differ by more than 0.2 g/L, repeat the test starting with two new 50 mL samples of filtered ground water.

5.5.5 ***Test report***

5.5.5.1 *Soil samples.* The test report shall state that the test was carried out in accordance with **5.5** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the acid-soluble sulphate content (if determined) to the nearest 0.01 % as (SO_3) of the oven-dry mass of soil passing a 2 mm test sieve;

c) the water-soluble sulphate content (if determined) either to the nearest 0.1 g/L or to the nearest 0.01 % (both as SO_3);

d) the percentage by dry mass of the original sample passing a 2 mm test sieve, to the nearest 1 %;

e) the information required by **9.1** of BS 1377-1:1990.

5.5.5.2 *Ground water samples.* The test report shall state that the test was carried out in accordance with **5.5** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the average sulphate content of the sample of ground water, to the nearest 0.01 g/L;

c) the information required by **9.1** of BS 1377-1:1990.

5.6 Ion-exchange method for analysis of water extract or ground water

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

NOTE The ion-exchange method cannot be used if the soil or ground water contains anions of strong acids, such as chlorides, nitrates and phosphates (see note **5.1.3**).

5.6.1 ***Apparatus***

5.6.1.1 *General.* The apparatus listed in **5.6.1.2** to **5.6.1.6** is required in addition to the items listed in **5.3.1** or **5.4.1**.

5.6.1.2 *Glass ion-exchange column,* fitted with a swan-neck outlet (see Figure 1).

5.6.1.3 *Water reservoir,* incorporating a constant-head device (optional). A typical design is shown in Figure 2.

5.6.1.4 *Two* 500 mL *conical beakers.*

5.6.1.5 250 mL *beaker.*

5.6.1.6 50 mL *burette and burette stand.*

5.6.1.7 *Amber-coloured glass container.*

5.6.2 ***Reagents***

5.6.2.1 *General.* All reagents shall be of recognized analytical reagent quality.

NOTE Where accurately standardized solutions are required it may be more convenient to obtain them already standardized in concentrated form and to dilute them as necessary in accordance with the manufacturer's instructions.

5.6.2.2 *A strongly acidic cation-exchange resin.*

5.6.2.3 *Hydrochloric acid solution* ($c(HCl)$ = approximately 4 mol/L). Dilute 360 mL of concentrated hydrochloric acid (density 1.18 g/mL) to 1 L with distilled water.

5.6.2.4 *Sodium hydroxide solution,* ($c(NaOH)$ = approximately 0.1 mol/L). Dissolve about 2 g of sodium hydroxide (see note 1) in 500 mL of distilled water. Determine its exact concentration (B) by titration against the potassium hydrogen phthalate solution (see **5.6.2.5**) using phenolphthalein or thymol blue as indicator (see note 2). Keep the solution in an airtight container.

NOTE 1 Sodium hydroxide is strongly caustic; care should therefore be taken to avoid contact with the skin, and eye protection is required.

NOTE 2 If standardized acid is available, e.g. prepared as described in **6.3.3**, this may be used instead of the potassium phthalate. Alternatively it may be more convenient to obtain a ready standardized solution of sodium hydroxide and prepare it in accordance with the manufacturer's instructions.

5.6.2.5 *Potassium hydrogen phthalate solution,* ($c(KHC_6H_4(COOH)_2)$ = 0.1 mol/L) (see note 2 to **5.6.2.4**). Weigh out 5.10 g of potassium hydrogen phthalate which has previously been dried at 110 °C for 2 h, dissolve in distilled water and dilute to exactly 250 mL in a volumetric flask.

5.6.2.6 *Indicator.* An indicator solution, e.g. screened methyl orange, which gives a distinct colour change in the range pH 4 to pH 5.

5.6.2.7 *Silver nitrate solution.* Dissolve 0.5 g of silver nitrate in 100 mL distilled water. Store the solution in an amber-coloured glass container.

5.6.2.8 *Nitric acid solution,* 5 % (v/v). Dilute 5 mL of concentrated nitric acid (density 1.42 g/mL) to 100 mL with distilled water.

5.6.2.9 *Distilled water,* of a purity complying with **5.1** of BS 1377-1:1990.

5.6.3 ***Preparation of ion-exchange column***

5.6.3.1 Place a quantity of cation-exchange resin sufficient to half-fill the column into a beaker and stir with distilled water.

5.6.3.2 Transfer the suspension of resin in water into the column so that when the resin has settled there is approximately 20 mm depth of water above the resin when the surplus water has drained away (see Figure 1). Maintain this depth of water above the resin at all times. Do not allow the resin to dry out.

5.6.3.3 If the constant head device is not available, activate the cation-exchange resin by leaching with 100 mL of hydrochloric acid (**5.6.2.3**) added in increments, followed by washing with distilled water in increments, taking care not to add further liquid until each increment has drained away.

5.6.3.4 Alternatively, if the constant-head device (Figure 2) is available, place 100 mL of the acid in the device, replace the stopper, and leave the apparatus until the acid has passed through the column. Then rinse the constant-head device, fill it with distilled water, and leave the water to percolate until the liquid coming from the column gives no turbidity when tested with about 1 mL of the silver nitrate solution acidified with a few drops of nitric acid.

5.6.3.5 After the column has been used for four consecutive determinations, re-activate the cation-exchange resin by repeating steps **5.6.3.3** and **5.6.3.4**.

5.6.4 *Test procedure*

5.6.4.1 Analyse each 2 : 1 water-soil extract obtained as described in **5.3.3.5**, or each ground water sample obtained as described in **5.4.2.4** as described in **5.6.4.2** to **5.6.4.6**.

5.6.4.2 Pass the solution through the ion-exchange column and rinse the column with two 75 mL increments of distilled water.

5.6.4.3 Collect the solution and washings in a 500 mL conical beaker placed under the outlet of the exchange column.

5.6.4.4 Add enough indicator to the contents of the conical beaker to impart sufficient colour for the detection of the end-point in **5.6.4.5**.

5.6.4.5 Titrate the solution against the standardized sodium hydroxide solution until the end-point is observed.

5.6.4.6 Record the volume (V) of sodium hydroxide solution required to neutralize the test solution to the nearest 0.05 mL.

5.6.5 *Calculations*

NOTE See form 3(d) of Appendix A.

5.6.5.1 Soil samples. If no other anions are present, the sulphate content can be expressed in terms of the concentration of sulphate in the extract (as SO_3) in g/L as in Method 1 below, or in terms of percentage (as SO_3) of water-soluble sulphate in the soil sample as in Method 2 below.

Method 1. Calculate the sulphate content in g/L of the 2 : 1 water/soil extract from the equation:

$$\text{Concentration of sulphate as } (SO_3) = 0.8BV \text{ g/L}$$

where

B is the concentration of the sodium hydroxide solution in mol/L

V is the volume of sodium hydroxide used in mL.

Method 2. Calculate the sulphate content as a percentage of the soil from the equation:

$$\text{Water-soluble sulphate content (as } SO_3) = 0.16BV\ (\%)$$

where

B is the concentration of the sodium hydroxide solution in mol/L

V is the volume of sodium hydroxide used in mL

5.6.5.2 *Ground water samples.* If no other anions are present calculate the sulphate content (as SO_3) in each test portion of the ground water (in g/L) from the equation:

$$SO_3 = 0.4\,B\,V$$

If individual results differ by no more than 0.2 g/L (SO_3), calculate the mean result. If they differ by more than 0.2 g/L, repeat the test starting with two new 100 mL samples of filtered ground water.

5.6.6 *Test report*

5.6.6.1 *Soil sample.* The test report shall state that the test was carried out in accordance with **5.6** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the average sulphate content (as SO_3) of the 2 : 1 water soil extract, to the nearest 0.01 g/L or the nearest 0.1 %;

c) the percentage by dry mass of the original sample passing a 2 mm test sieve, to the nearest 1 %;

d) the information required by **9.1** of BS 1377-1:1990.

5.6.6.2 *Ground water sample.* The test report shall state that the test was carried out in accordance with **5.6** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the average sulphate content of the sample of ground water, to the nearest 0.01 g/L;

c) the information required by **9.1** of BS 1377-1:1990.

6 Determination of the carbonate content

6.1 General

This clause describes two tests for the determination of the carbonate content of a soil. Both tests depend upon the reaction between carbonates and hydrochloric acid, which liberates carbon dioxide. The first is a rapid titration method which is suitable for soils in which the carbonates exceed 10 % (m/m), and where an accuracy of about 1 % is sufficient. The second uses the gravimetric method described for hardened concrete in BS 1881-124, to which reference is made. The initial preparation of test specimens is similar for both methods.

NOTE Good practice in chemical testing requires that duplicate specimens should be tested. In each of the test methods the measurement of only one value of the overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a mean value and an indication of the reliability of the results. Guidance on the number of measurements required and the treatment of the results obtained is beyond the scope of this Part of this standard.

In the rapid titration method the soil specimen is treated with a known quantity of acid to finality. The amount of excess acid is determined by titration against sodium hydroxide. The result is calculated in terms of the equivalent proportion of carbon dioxide.

In the gravimetric method the carbon dioxide evolved when the soil is treated with hydrochloric acid is passed through a granular absorbent which enables the mass of carbon dioxide to be determined gravimetrically.

6.2 Preparation of sample

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

6.2.1 ***Apparatus***

6.2.1.1 *Drying oven,* capable of being controlled to maintain a temperature range of 75 °C to 80 °C, as well as the range 105 °C to 110 °C.

6.2.1.2 *Balance,* readable to 0.001 g.

6.2.1.3 *Desiccator,* containing anhydrous silica gel.

6.2.1.4 *Test sieves,* 2 mm and 425 µm aperture sizes, with receiver.

6.2.1.5 *Pestle and mortar,* or a suitable mechanical crusher.

6.2.1.6 *Sample dividers of multiple-slot type (riffle boxes),* having widths of opening of 7 mm and 15 mm.

6.2.1.7 *Glass weighing bottle,* approximately 50 mm diameter, 25 mm high and fitted with a ground glass stopper.

6.2.1.8 *Glass or plastics funnel,* about 100 mm diameter.

6.2.2 ***Preparation of soil sample***

6.2.2.1 Obtain an initial sample as described in **7.3**, and of the appropriate size specified in **7.5** of BS 1377-1:1990.

6.2.2.2 Dry this sample in an oven at 105 °C to 110°C and, cool to room temperature in the desiccator.

6.2.2.3 Sieve the sample on a 2 mm test sieve (if appropriate, guarded by a sieve of a larger aperture).

6.2.2.4 Crush all retained particles to pass the 2 mm sieve and mix thoroughly with the material already passing the sieve.

6.2.2.5 Divide the material passing the 2 mm sieve by successive riffling through the 15 mm divider to produce a representative sample of about 50 g.

6.2.2.6 Sieve this sample on a 425 µm test sieve.

6.2.2.7 Crush all retained particles to pass the 425 µm test sieve, and mix thoroughly with the material already passing the sieve.

6.2.2.8 Use this sample for preparing the specimens for testing (see **6.3.5** or **6.4.3**).

6.3 Rapid titration method

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

6.3.1 ***Apparatus***

6.3.1.1 250 mL *tall-form beaker and watch glass cover.*

6.3.1.2 *Two* 100 mL *burettes* reading to 0.1 mL.

6.3.1.3 25 mL *pipette.*

6.3.1.4 250 mL *conical flask.*

6.3.1.5 1L *volumetric flask.*

6.3.2 ***Reagents***

6.3.2.1 *General.* All reagents shall be of recognized analytical reagent quality.

NOTE Where accurately standardized solutions are required it may be more convenient to obtain them already standardized in concentrated form and to dilute them as necessary in accordance with the manufacturer's instructions.

6.3.2.2 *Hydrochloric acid* (c(H Cl) = approximately 1 mol/L). Dilute 88 mL of concentrated hydrochloric acid with distilled water to make 1 L of solution.

6.3.2.3 *Sodium hydroxide solution* (c(NaOH) = approximately 1 mol/L). Dissolve about 20 g of sodium hydroxide in 500 mL of distilled water and store in an airtight plastic container. Determine the concentration (B) of this solution by preparing a 0.1B diluted solution (pipette 25 mL of the concentrated solution into a 250 mL volumetric flask and dilute to 250 mL with distilled water). Determine the concentration (B/10) of the diluted solution as described in **5.6.2.4** and multiply by 10 to obtain the concentration (B) of the concentrated solution.

NOTE Sodium hydroxide is strongly caustic; care should therefore be taken to avoid contact with the skin and eye protection is required.

6.3.2.4 *Screened methyl orange indicator.*

NOTE Screened methyl orange gives a more distinct end-point than the unscreened indicator but the latter may be used if preferred. Methyl red or bromcresol green are also suitable.

6.3.3 *Standardization of the hydrochloric acid*

6.3.3.1 Pipette 25 mL of the hydrochloric acid into a 250 mL conical flask.

6.3.3.2 Place the conical flask on a white background and add the sodium hydroxide solution slowly from a burette (see note). During this operation, rotate the flask constantly with one hand while controlling the stopcock on the burette with the other.

NOTE Sodium hydroxide should not remain in the burette for prolonged periods and after use the burette should be thoroughly rinsed with water. Failure to carry out these instructions could damage the burette and cause the tap to seize.

6.3.3.3 Continue adding sodium hydroxide until the acid is neutralized.

6.3.3.4 Record the volume of sodium hydroxide used.

6.3.3.5 Repeat steps **6.3.4.1** to **6.3.4.4** using two more 25 mL aliquots of acid solution. The volumes of sodium hydroxide used for each titration shall not differ by more than 0.1 mL.

6.3.3.6 Calculate the mean volume of sodium hydroxide used V_1 (in mL) and calculate the concentration (H) of the hydrochloric acid solution (in mol/L) from the following equation:

$$H = \frac{V_1\,B}{25}$$

where B is the concentration of the sodium hydroxide solution (in mol/L).

6.3.4 *Preparation of test specimen*

6.3.4.1 From the sample prepared as in **6.2.2** take a representative portion containing a little over 5 g of oven-dry soil.

6.3.4.2 Dry this portion in the oven at 105 °C to 110 °C. The specimen is deemed to be dry when the difference in successive weighings at intervals of 4 h do not exceed 0.1 % of the specimen mass.

6.3.4.3 Allow the specimen to cool to room temperature in the desiccator.

6.3.4.4 Weigh out about 5 g of dry soil as the test specimen and record its mass (m) to the nearest 0.001 g.

6.3.5 *Analysis of test specimen*

6.3.5.1 Place the weighed specimen into the 250 mL tall-form beaker.

6.3.5.2 Add 100 mL of the hydrochloric acid solution slowly from the burette.

6.3.5.3 Cover the beaker with the watch glass and allow to stand for 1 h, stirring occasionally.

6.3.5.4 When the soil has settled after the final stirring, remove 25 mL of the supernatant liquid with the pipette and transfer to a conical flask.

6.3.5.5 Add six drops of the indicator solution and titrate with the sodium hydroxide solution as described in **6.3.3** until the same colour change as was observed in the standardization procedure (**6.3.3.3**) occurs. Record the volume (V_2) of sodium hydroxide solution used, to the nearest 0.1 mL.

6.3.6 *Calculations*

NOTE See form 3(e) of Appendix A.

Calculate the carbonate content of the soil, as a percentage of CO_2, from the equation:

$$\text{Carbonate (as CO}_2\text{)} = \frac{8.8\ (25H - BV_2)}{m}$$

where

H is the concentration of the hydrochloric acid (in mol/L);

B is the concentration of the sodium hydroxide solution (in mol/L);

m is the mass of the soil specimen (in g);

V_2 is the volume of sodium hydroxide used (in mL).

6.3.7 *Test report.* The test report shall state that the test was carried out in accordance with **6.3** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the percentage carbonates in the soil sample, expressesd as CO_2, to two significant figures;

c) the information required by **9.1** of BS 1377-1:1990.

6.4 Gravimetric method

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

6.4.1 ***Apparatus***

6.4.1.1 *General.* Apparatus as described in **5.3** of BS 1881-124:1988.

6.4.1.2 *Balance,* readable to 0.0001 g.

6.4.2 *Reagents.* The reagents are as described in **5.2.13** of BS 1881-124:1988.

6.4.3 ***Preparation of test specimen***

6.4.3.1 From the sample prepared as described in **6.2.2** take a representative test specimen of the required mass.

NOTE As an approximate guide, the amount of soil required will range from about 0.2 g for a pure limestone or chalk to 1 g for a relatively non-calcareous soil. If in doubt the appropriate quantity should be ascertained from preliminary trial tests.

6.4.3.2 Dry the specimen in the oven at 105 °C to 110 °C. The specimen is deemed to be dry when the difference in successive weighings at intervals of 4 h do not exceed 0.1 % of the specimen mass.

6.4.3.3 Allow the specimen to cool to room temperature in the desiccator.

6.4.3.4 Determine the mass of the specimen to 0.0001 g.

6.4.4 *Analysis of test specimen and calculations.* Proceed as described in **5.7** of BS 1881-124:1988.

6.4.5 *Test report.* The test report shall state that the test was carried out in accordance with **6.4** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the percentage carbonate in the soil sample, expressed as CO_2;

c) the information required by **9.1** of BS 1377-1:1990.

7 Determination of the chloride content

7.1 General

7.1.1 *Principle.* This clause decribes two tests for the determination of water-soluble and acid-soluble chloride salt content of soil. Both procedures are based on Volhard's method.

NOTE The principle can also be used for determining chlorides in ground water for which Mohr's method is more suitable, see BRE Current Paper CP 2/79, published by Building Research Establishment, Garston, Watford, Herts.

For the determination of water-soluble chlorides (see **7.2**) the chlorides are extracted from a dry soil sample by solution in a mass of water equal to twice that of the sample. Results are expressed as the chloride ion content.

A qualitative test for checking for the presence of chlorides is included, which if negative obviates the need for the quantitative analysis.

For the determination of acid-soluble chlorides (see **7.3**), which includes chlorides not extracted by water, chlorides are extracted from a dry soil sample with dilute nitric acid.

Results are expressed as the chloride content.

NOTE Good practice in chemical testing requires that duplicate specimens should be tested. In each of the test methods the measurement of only one value of the overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a mean value and an indication of the reliability of the results. Guidance on the number of measurements required and the treatment of the results obtained is beyond the scope of this Part of this standard.

7.1.2 *Applications.* The water-extract method is applicable only to soils in which the chloride content derives directly from recent contact with, or immersion in, saline water.

NOTE The principle can also be used for determining chlorides in ground water for which Mohr's method is more suitable, see BRE Current Paper CP 2/79, published by Building Research Establishment, Garston, Watford, Herts.

The acid-extract method is applicable to the determination of the chloride content of soils from desert areas or where the origin of the chlorides is uncertain.

7.2 Determination of water-soluble chloride content

The requirements of Part 1 of the standard, where appropriate, shall apply to this test method.

7.2.1 ***Apparatus***

7.2.1.1 *Balance,* readable to 1 g.

7.2.1.2 *Balance,* readable to 0.001 g.

7.2.1.3 1L *volumetric flask.*

7.2.1.4 10 mL *graduated glass measuring cylinder.*

7.2.1.5 500 mL *graduated glass measuring cylinder.*

7.2.1.6 100 mL *pipette.*

7.2.1.7 25 mL *pipette.*

7.2.1.8 *Two* 50 mL *burettes.*

7.2.1.9 *Stoppered conical flasks,* 250 mL capacity. (At least four.)

7.2.1.10 *Wash bottle,* preferably made of plastics, containing distilled water.

7.2.1.11 *Amber-coloured glass reagent bottle.*

7.2.1.12 *Wide mouth screw-capped plastics or metal bottle,* of 2 L capacity. The cap shall be watertight when closed.

7.2.1.13 *Mechanical shaking apparatus,* capable of keeping 500 g of soil and 1 000 mL of water contained in the bottles in continuous suspension.

NOTE A device which rotates the containers end over end at 30 r/min to 60 r/min is satisfactory. Shaking machines giving a vibrating motion are also suitable.

7.2.1.14 *Drying oven,* capable of being controlled to maintain temperatures of 105 ± 5 °C and 150 ± 5 °C, complying with **4.2.2.1** of BS 1377-1:1990.

7.2.1.15 *Desiccator containing anhydrous silica gel.*

7.2.1.16 *Filter funnel,* of approximately 100 mm diameter.

7.2.1.17 *Filter papers,* of a diameter appropriate to the size of the funnel: medium grade (e.g. Whatman No. 40) and fine grade (e.g. Whatman No. 42).

7.2.1.18 *Test sieve,* of 2 mm aperture size, with receiver.

7.2.1.19 *Sample dividers of multiple-slot type (riffle boxes),* complying with **7.2.1.7** of BS 1377-1:1990 having widths of opening of 7 mm and 15 mm.

7.2.1.20 *Pestle and mortar,* or a suitable mechanical crusher.

7.2.1.21 500 mL *volumetric flask.*

7.2.1.22 *Two beakers,* of about 250 mL capacity.

7.2.2 *Reagents*

7.2.2.1 *General.* All reagents shall be of recognized analytical reagent quality.

NOTE Where accurately standardized solutions are required it may be more convenient to obtain them already standardized in concentrated form and to dilute them as necessary in accordance with the manufacturer's instructions.

7.2.2.2 *Silver nitrate solution* ($c(A_gNO_3)$ = 0.100 mol/L). Dry about 20 g of silver nitrate at not more than 150 °C for 1 h to 2 h and allow to cool in the desiccator. Weigh out 16.987 g of the dried silver nitrate, dissolve in distilled water and dilute to 1 L in a volumetric flask. Store the solution in the amber-coloured glass reagent bottle and protect from prolonged exposure to sunlight.

7.2.2.3 *Thiocyanate solution,* ($c(NH_4SCN)$ or $c(KSCN)$ = approximately 0.1 mol/L). Dissolve 10.5 g of potassium thiocyanate or 8.5 g of ammonium thiocyanate in distilled water to 1 L in a volumetric flask.

7.2.2.4 *Nitric acid solution,* ($c(HNO_3)$ = approximately 6 mol/L). Dilute 100 mL of nitric acid (70 % HNO_3 1.42 g/mL) with distilled water to 250 mL and boil the diluted acid until it is colourless.

7.2.2.5 *3,5,5-Trimethylhexan-1-ol.*

7.2.2.6 *Ferric alum indicator solution.* Add 60 g of water to 75 g of ammonium ferric sulphate, warm to dissolve, and add 10 mL of nitric acid (**7.2.2.4**). Allow to cool and store in a glass bottle.

7.2.3 *Test procedure*

7.2.3.1 *Standardization of thicyanate solution*

7.2.3.1.1 Determine the concentration of the thiocyanate solution as given in **7.2.3.1.2** to **7.2.3.1.5**.

7.2.3.1.2 Transfer 25 mL of the silver nitrate solution into a 250 mL conical flask, using a pipette, and add 5 mL of the nitric acid solution and 1 mL of ferric alum indicator solution.

7.2.3.1.3 Add thiocyanate solution from a burette until the first permanent colour change occurs, that is from colourless to pink.

7.2.3.1.4 Record the volume of thiocyanate solution added V_1 (in mL).

7.2.3.1.5 Calculate the concentration C (in mol/L) of the solution from the following equation:

$$C = \frac{2.5}{V_1}$$

7.2.3.2 *Preparation of test specimen*

7.2.3.2.1 Prepare a specimen for analysis from the laboratory sample as given in **7.2.3.2.2** to **7.2.3.2.8**.

7.2.3.2.2 Obtain an initial sample as described in **7.3**, and of the approximate size specified in **7.5**, of BS 1377-1:1990.

7.2.3.2.3 Dry this sample in an oven at 105 °C to 110 °C, and allow to cool to room temperature in the desiccator.

7.2.3.2.4 Sieve the sample on a 2 mm test sieve (if appropriate, guarded by a test sieve of larger aperture) and crush retained particles other than stones to pass through the 2 mm test sieve.

7.2.3.2.5 Reject the stones, ensuring that no fine material adheres to them, e.g. by brushing. Record the mass m_2 (in g) of sample passing the 2 mm test sieve to the nearest 0.1 %. Throughout these and subsequent operations ensure that there is no loss of fines.

7.2.3.2.6 Divide the material passing the 2 mm test sieve by successive riffling through the 15 mm divider to produce the following:

a) a test specimen of about 500 g; and

b) a specimen of about 50 g for a qualitative check test to determine whether chlorides are present.

7.2.3.2.7 Dry the specimens in the oven at 105 °C to 110 °C. The specimens are deemed to be dry when the differences in successive weighings carried out at intervals of 4 h, do not exceed 0.1 % of the original mass of the sample.

7.2.3.2.8 Allow the specimens to cool to room temperature in the desiccator.

7.2.3.3 *Qualitative check for chlorides*

7.2.3.3.1 Verify the presence of chlorides in the soil as described in **7.2.3.3.2** to **7.2.3.3.6**.

7.2.3.3.2 Place the 50 g check specimen in a 500 mL conical flask and add to it an approximately equal mass of distilled water.

7.2.3.3.3 Agitate the contents intermittently for 4 h, allow to settle and pour off some of the supernatant solution into a beaker.

7.2.3.3.4 Obtain about 25 mL of clear solution by filtering, if necessary, through a medium grade filter paper, e.g. Whatman No. 40.

7.2.3.3.5 Acidify the liquid with nitric acid, add about five drops of the silver nitrate solution and allow to stand for 10 min.

7.2.3.3.6 If no turbidity is apparent after this time the soluble chloride ion content of the soil is negligible and the test for chloride content is not necessary.

7.2.3.4 *Preparation of water-soluble chloride extract*

7.2.3.4.1 Obtain the water-soluble chloride extract from each test portion as described in **7.2.3.4.2** to **7.2.3.4.7**.

7.2.3.4.2 Weigh a clean and dry screw-capped bottle to 1 g, and record its mass.

7.2.3.4.3 Place the test specimen in the bottle and weigh the bottle and contents to 1 g.

7.2.3.4.4 Calculate the mass of soil by difference m (in g).

7.2.3.4.5 Add to the bottle a mass of distilled water m_w equal to twice the mass of the test specimen.

NOTE A portion of the same 2 : 1 water-soil extract as prepared for sulphate analysis may be used. However for non-cohesive soils a 1 : 1 extract may be more convenient, in which case a different factor is used for the calculation in **7.2.4**.

Fasten the watertight caps securely.

7.2.3.4.6 Secure the bottle to the shaking apparatus and shake for at least 16 h. When convenient the soil can be left shaking overnight.

7.2.3.4.7 Filter the suspension through a medium-grade filter paper into a clean beaker until at least 100 mL of clear filtrate has been collected. If the filtrate is not completely clear, filter through a fine-grade filter paper.

NOTE If the solids settle quickly and the supernatant liquid is clear it can be carefully poured off instead of filtering.

7.2.3.5 *Analysis of extract*

7.2.3.5.1 Analyse each water extract sample as described in **7.2.3.5.2** to **7.2.3.5.8**.

7.2.3.5.2 Take 100 mL of the filtered extract by means of the pipette and transfer to the 250 mL conical flask.

7.2.3.5.3 Add 5 mL of the nitric acid solution to the flask followed by silver nitrate solution from a burette until all the chloride has been precipitated, and then add a little excess silver nitrate.

7.2.3.5.4 Record the total volume V (in mL) of silver nitrate solution added.

7.2.3.5.5 Add 2 mL of 3,5,5-trimethylhexan-1-ol, fit the stopper, and shake the flask vigorously to coagulate the precipitate.

7.2.3.5.6 Carefully loosen the stopper, avoiding loss of solution, rinse with distilled water, and collect the washings in the solution.

7.2.3.5.7 Add 5 mL of the ferric alum indicator solution, followed by the standardized thiocyanate solution from a burette until the first permanent colour change occurs, that is from colourless to brick-red, and is the same depth of colour as was used for the standardization described in **7.2.3.1**.

7.2.3.5.8 Record the volume V_3 (in mL) of thiocyanate solution added.

7.2.4 *Calculations*

NOTE See form 3(f) of Appendix A.

Calculate the amount of chloride ions present in each water extract, as a percentage by dry mass of soil, from the equation:

$$\text{chloride ion content} = 0.007092\ (V_2 - 10\ CV_3)$$

where

V_2 is the volume of the silver nitrate solution added (in mL);

V_3 is the volume of the standardized thiocyanate solution added (in mL);

C is the concentration of the standardized thiocyanate solution (in mol/L)

NOTE If the water-soil ratio is 1 : 1 instead of 2 : 1 the constant 0.007092 in this equation is replaced by 0.003546.

If more than one specimen has been tested, and if the individual results differ by no more than 0.1 % of chloride ion content, calculate the mean result. If they differ by more than 0.1 %, repeat the test starting with two new test specimens.

7.2.5 *Test report.* The test report shall state that the test was carried out in accordance with **7.2** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the percentage of chloride ions in the soil sample, to the nearest 0.01 %;

c) the water to soil ratio used for preparing the soluble extract;

d) the information required by **9.1** of BS 1377-1:1990

7.3 Determination of acid-soluble chloride content

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

7.3.1 ***Apparatus***

7.3.1.1 *Balance* readable to 1 g.

7.3.1.2 *Balance,* readable to 0.001 g.

7.3.1.3 1 L *volumetric flask.*

7.3.1.4 10 mL *graduated glass measuring cylinder.*

7.3.1.5 50 mL *graduated glass measuring cylinder.*

7.3.1.6 10 mL *graduated glass measuring cylinder.*

7.3.1.7 15 mL *pipette.*

7.3.1.8 500 mL *beaker.*

7.3.1.9 *Two* 50 mL *burettes.*

7.3.1.10 *Stoppered conical flasks,* 250 mL capacity. (At least two.)

7.3.1.11 *Filter funnel,* of approximately 100 mm diameter.

7.3.1.12 *Filter papers,* of a diameter appropriate to the size of the funnel: coarse grade (e.g. Whatman No. 541).

7.3.1.13 *Test sieve,* of 150 µm aperture size with receiver.

7.3.1.14 *Pestle and mortar,* or a suitable mechanical crusher.

7.3.1.15 *Sample dividers of multiple-slot type (riffle boxes).*

7.3.2 *Reagents.* The reagents required are the same as those listed in **7.2.2**.

7.3.3 ***Test procedure***

7.3.3.1 *Standardization of potassium thiocyanate solution.* Follow the procedure described in **7.2.3.1**.

7.3.3.2 *Preparation of test specimen*

7.3.3.2.1 Prepare a specimen for analysis from the laboratory sample as described in **7.3.3.2.2** to **7.3.3.2.7**.

7.3.3.2.2 Obtain an initial sample as described in **7.3**, and of the approximate size specified in **7.5**, of BS 1377-1:1990.

7.3.3.2.3 Dry this sample in an oven at 105 °C to 110 °C, and cool to room temperature in the desiccator.

7.3.3.2.4 Sieve the sample on a 150 µm test sieve (if appropriate, guarded by a sieve of larger aperture).

7.3.3.2.5 Crush all retained particles to pass the 150 μm sieve and mix thoroughly with the material already passing the sieve.

7.3.3.2.6 Divide the material by successive riffling to produce test specimens each of about 10 g.

7.3.3.2.7 Dry the test specimens in the oven at 105 °C to 110 °C, and allow to cool in the desiccator.

7.3.3.3 *Preparation of acid-soluble chloride extract*

7.3.3.3.1 Obtain the acid-soluble chloride extract from each test portion as described in **7.3.3.3.2** to **7.3.3.3.11**.

7.3.3.3.2 Weigh out 5 ± 0.005 g of the test portion and place it in a 500 mL beaker.

7.3.3.3.3 Add 50 mL of distilled water to disperse the particles, followed by 15 mL of nitric acid.

7.3.3.3.4 Heat to near boiling point and keep warm for 10 min to 15 min.

7.3.3.3.5 Filter through a coarse-grade filter paper into a conical flask, wash with hot water and collect the washings with the filtrate.

NOTE Some cloudiness in the filtrate is acceptable.

7.3.3.3.6 Allow to cool, and add silver nitrate solution from a burette until all the chloride has been precipitated, then add a little excess silver nitrate.

7.3.3.3.7 Record the total volume V_2 (in mL) of silver nitrate solution added.

7.3.3.3.8 Add 2 mL of 3,5,5-trimethylhexan-1-ol, fit the stopper, and shake the flask vigorously to coagulate the precipitate.

7.3.3.3.9 Carefully loosen the stopper, avoiding loss of solution, rinse with distilled water, and collect the washings in the solution.

7.3.3.3.10 Add 5 mL of the ferric alum indicator solution, followed by the standardized potassium thiocyanate solution from a burette until the first permanent colour change occurs, that is from colourless to brick-red, and is the same depth of colour as was used for the standardization described in **7.2.3.1**.

7.3.3.3.11 Record the volume V_3 (in mL) of thiocyanate solution added.

7.3.4 ***Calculations***

NOTE See form 3(f) of Appendix A.

Calculate the chloride content of the soil as a percentage by dry mass of soil from the equation:

$$\text{chloride content} = 0.07092\,(V_2 - 10CV_3)$$

where

V_2 is the volume of the silver nitrate solution added (in mL);

V_3 is the volume of standardized thiocyanate solution added (in mL);

C is the concentration of the standardized thiocyanate solution, (in mol/L).

If more than one specimen has been tested, and if the results differ by no more than 0.1 % of chloride content, calculate the mean result. If they differ by more than 0.1 %, repeat the test starting with two new test specimens.

7.3.5 *Test report.* The test report shall state that the test was carried out in accordance with **7.3** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the percentage of chlorides in the soil sample, to the nearest 0.01 %;

c) the method used, i.e. the acid extraction method;

d) the information required by **9.1** of BS 1377-1:1990.

8 Determination of total dissolved solids

8.1 General

This clause describes a method for determining the total amount of dissolved solids in a sample of water, e.g. ground water.

NOTE Good practice in chemical testing requires that duplicate specimens should be tested. In each of the test methods the measurement of only one value of the overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a mean value and an indication of the reliability of the results. Guidance on the number of measurements required and the treatment of the results obtained is beyond the scope of this Part of this standard.

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

8.2 Apparatus

8.2.1 *Buchner funnel,* of about 100 mm diameter.

8.2.2 *Vacuum filtration flask,* of about 500 mL capacity, to take the funnel.

8.2.3 *Source of vacuum,* and vacuum tubing.

8.2.4 *Filter papers,* to fit the funnels, e.g. Whatman No. 40.

8.2.5 An *evaporating dish,* which can be accommodated on the balance described in **8.2.9**.

8.2.6 *Drying oven,* capable of maintaining a temperature of 180 °C to within ± 10 °C.

8.2.7 *Desiccator,* containing anhydrous silica gel.

8.2.8 *Volumetric flask.*

NOTE The size of the flask depends on the amount of water required for test (see **8.3.2**).

8.2.9 *Balance* readable to 0.5 mg.

8.2.10 *Electric hotplate,* or Bunsen burner and tripod.

8.2.11 *Shallow container,* suitable for use as a boiling water bath.

8.2.12 *Wash-bottle,* preferably made of plastics, containing distilled water.

8.3 Test procedure

8.3.1 Filter the sample of water to remove any suspended solids, using the Buchner funnel and flask.

8.3.2 Collect a known volume V (in mL) of filtered water (sufficient to yield between 2.5 mg and 1 000 mg of dissolved solids) in the volumetric flask.

8.3.3 Heat an evaporating dish in the oven at 180 ± 10 °C for 30 min, allow to cool in the desiccator and determine its mass m_1 to 0.0005 g.

8.3.4 Pour a portion of the water sample into the evaporating dish and evaporate on the boiling water bath. Do this in a clean atmosphere to prevent contamination by airborne solids.

8.3.5 Add further portions of the water sample to the dish as evaporation proceeds. When the flask is empty rinse it twice with 10 mL of distilled water and add the rinsings to the evaporating dish.

8.3.6 Allow to evaporate to dryness and wipe dry the outside of the dish.

8.3.7 Place the evaporating dish and contents into the oven and heat at 180 ± 10 °C for 1 h.

8.3.8 Allow the dish to cool in the desiccator to room temperature, and determine its mass m_2 to 0.0005 g.

8.3.9 Repeat steps **8.3.7** and **8.3.8**, but heating in the oven for 30 min, until the difference between successive weighings does not exceed 1 mg.

8.4 Calculations

Calculate the total dissolved solids (TDS) (in mg/L) in each measured sample of water from the equation:

$$\mathrm{TDS} = \frac{m_2 - m_1}{V} \times 10^6$$

where

m_1 is the mass of the dried evaporating dish (in g);

m_2 is the mass of the dish with dissolved solids after the second or subsequent period of drying at 180 °C (in g);

V is the measured volume of the sample of water used (in mL).

Calculate the mean of the individual results if more than one sample has been tested. If they differ by more than 10 % of the mean value, repeat the test starting with two new water samples.

8.5 Test report

The test report shall state that the test was carried out in accordance with clause **8** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the total dissolved solids (TDS) (in mg/L), in the water sample, to two significant figures;

c) whether it was possible to filter the water sample free of all traces of turbidity;

d) information about the origin of the water sample;

e) the information required by **9.1** of BS 1377-1:1990.

9 Determination of the pH value

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

9.1 General

This clause describes the procedure for determining the pH value, by the electrometric method, which gives a direct reading of the pH value of a soil suspension in water. This method can also be used for determining the pH value of a sample of ground water.

NOTE Good practice in chemical testing requires that duplicate specimens should be tested. In each of the test methods the measurement of only one value of the overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a mean value and an indication of the realiability of the results. Guidance on the number of measurements required and the treatment of the results obtained is beyond the scope of this Part of this standard.

9.2 Apparatus

9.2.1 *Apparatus for preparation of test specimens*

9.2.1.1 *Balance,* readable to 0.001 g.

9.2.1.2 *Pestle and mortar,* or a suitable mechanical crusher.

9.2.1.3 *Test sieve,* of 2 mm aperture size, with receiver.

9.2.1.4 *Non-corrodible tray.*

9.2.2 *Apparatus for electrometric method of pH determination*

9.2.2.1 *pH meter,* fitted with a glass electrode and a calomel reference electrode (which may be incorporated in one probe) covering the range pH 3.0 to pH 10.0. The scale shall be readable and accurate to 0.05 pH units.

9.2.2.2 *Three* 100 mL *glass or plastics beakers with cover glasses and stirring rods.*

9.2.2.3 *Two* 500 mL *volumetric flasks.*

9.2.2.4 *Wash bottle,* preferably made of plastics, containing distilled water.

9.3 Reagents

9.3.1 *General.* All reagents shall be of recognized analytical reagent quality.

9.3.2 *Buffer solution,* pH 4.0. Dissolve 5.106 g of potassium hydrogen phthalate in distilled water and dilute to 500 mL with distilled water. Alternatively, a proprietary buffer solution of pH 4.0 may be used.

9.3.3 *Buffer solution,* pH 9.2. Dissolve 9.54 g of sodium tetraborate (borax) in distilled water and dilute to 500 mL. Alternatively, a proprietary buffer solution of pH 9.2 may be used.

9.3.4 *Potassium chloride.* Saturated solution (for maintenance of the calomel electrode).

9.4 Preparation of test specimen

9.4.1 Obtain an initial sample as described in **7.3**, and of the appropriate size specified in **7.5** of BS 1377-1:1990.

9.4.2 Allow the sample to air-dry by spreading out on a tray exposed to air at room temperature.

9.4.3 Sieve the sample on a 2 mm test sieve (if appropriate, guarded by a sieve of larger aperture) and crush retained particles other than stones to pass through the 2 mm test sieve.

9.4.4 Reject the stones, ensuring that no fine material adheres to them, e.g. by brushing. Record the mass m_2 (in g) of the sample passing the 2 mm test sieve to the nearest 0.1 %. Throughout these and subsequent operations ensure that there is no loss of fines.

9.4.5 Divide the material passing the 2 mm test sieve by successive riffling through the 15 mm divider to produce a representative test sample of 30 g to 35 g.

9.5 Electrometric method of pH determination

9.5.1 From the sample obtained as described in **9.4**, weigh out 30 ± 0.1 g of soil and place in a 100 mL beaker.

9.5.2 Add 75 mL of distilled water to the beaker, stir the suspension for a few minutes, cover with a cover glass and allow to stand for at least 8 h.

NOTE The pH value of a soil suspension varies with the ratio of soil to water, an increase in dilution bringing the pH closer to 7.

9.5.3 Stir the suspension again immediately before testing.

9.5.4 Calibrate the pH meter by using the standard buffer solutions, following the procedure recommended by the manufacturer.

9.5.5 Wash the electrode with distilled water and immerse in the soil suspension. Take two or three readings of the pH of the suspension with brief stirrings between each reading. These readings shall agree to within 0.05 pH units before being accepted.

NOTE The pH readings of the soil suspension should reach a constant value in about 1 min. No readings should be taken until the pH meter has reached equilibrium.

9.5.6 Remove the electrodes from the suspension and wash them with distilled water. Re-check the calibration of the pH meter against one of the standard buffer solutions.

9.5.7 If the instrument is out of adjustment by more than 0.05 pH units, set it to the correct adjustment and repeat steps **9.5.5** and **9.5.6** until consistent readings are obtained.

9.5.8 When not in use, leave the electrode standing in a beaker of distilled water.

9.6 Test Report

The test report shall state that the test was caried out in accordance with **9.5** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the pH value of the soil suspension to the nearest 0.1 pH unit;

c) the information required by **9.1** of BS 1377-1:1990.

10 Determination of resistivity

10.1 General

10.1.1 *Principle.* The tests described in this clause are for the determination of the electrical resistivity of samples of soil in the laboratory. The resistivity value indicates the relative capability of the soil to carry electric currents from which the corrosiveness of the soil can be deduced.

10.1.2 *Types of test.* Three types of test are described. In the first, disc electrodes are fitted to the end of a cylindrical undisturbed or compacted soil sample. A voltage is applied across the electrodes, the current flowing between them is measured and the application of Ohm's law enables the electrical resistance to be derived from which the soil resistivity is calculated.

In the second method four probe electrodes are inserted into the sample in a Wenner configuration. A current is passed between the outer electrodes and the resistance between the two inner electrodes is determined by balancing a bridge circuit, from which the soil resistivity is calculated. This is the same principle as used for the in-situ test described in **5.1** of BS 1377-9:1990.

The third method is similar to the first except that it applies to free-draining soil which is compacted into a rectangular box fitted with end electrodes, and is flooded with water before applying the voltage.

10.1.3 *Limitations.* Soil samples that have been compacted in the laboratory contain a significant proportion of air voids, unless the moisture content is appreciably higher than the optimum for the applied compactive effort. These air voids cause high resistances and can restrict contact between the soil and the electrodes. These effects can be reduced by a thorough mixing of the soil to prevent segregation, but in some instances it may be better to use only the fine fraction of the soil, e.g. the material passing a 2 mm test sieve.

Laboratory tests do not take account of changes in the soil characteristics which take place with time, unless further tests are carried out at suitable intervals.

The test procedures as described for compacted soils are for single sets of measurements obtained at a particular moisture content. There may be instances where further tests should be carried out over a range of moisture contents (and if appropriate over a range of densities) so that the conditions of interest can be bracketed.

The results of these tests should be interpreted by a specialist.

10.2 Measurement of resistivity: disc electrodes method

10.2.1 *General.*This procedure enables the electrical resistivity of an undisturbed or disturbed sample of soil to be measured in the laboratory. It is the preferred method for soils containing gravel-size particles. An undisturbed sample may be obtained in a sampling tube from a borehole, or it may be prepared from a block sample taken from an open excavation.

A disturbed sample may be compacted in the laboratory to a specified dry density at a specified moisture content. The moisture content may be the natural in-situ moisture content, or it may be related to the optimum moisture content for a certain degree of compaction if the relevant moisture-density relationship for the soil has already been determined.

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

10.2.2 ***Apparatus***

10.2.2.1 *Rigid cylindrical container,* (referred to as the test container) of non-conducting material at least 100 mm internal diameter and 400 mm to 1 000 mm long. The sampling tube may be used if it is of suitable material.

NOTE Rigid plastics piping of PVC or polypropylene have been found to be suitable.

10.2.2.2 *Suitable means of extruding the sample* from the sampling tube and into the test container, such as that referred to in **8.2.2** of BS 1377-1:1990. This is used when an undisturbed sample is not received in a suitable non-conducting container.

10.2.2.3 *Two metal discs,* of a diameter about 1 mm less than that of the test container, to act as electrodes, each having a recessed terminal for connecting to insulated copper wire.

10.2.2.4 *d.c. power source*, a calibrated voltmeter and a calibrated low-resistance ammeter.

NOTE If a test is unduly prolonged or if the electrodes are not properly cleaned before use a d.c. system can cause polarization effects in soil leading to a resistivity higher than the actual value being measured, giving an error on the unsafe side.

The instruments shall have a performance complying with class **2.5** of BS 89, and shall be re-calibrated at intervals not exceeding 2 years.

10.2.2.5 *Insulated copper* wire, for connecting the electrodes to the resistivity meter, fitted at each end with suitable terminals to ensure good electrical contact.

NOTE Standard copper wire of about 1.5 mm^2 cross-sectional area is suitable.

10.2.2.6 *Apparatus for the determination of moisture content,* as decribed in **3.2** of BS 1377-2:1990.

10.2.2.7 *Sample trimming tools,* e.g. trimming knife, spatula, end-trimming tool.

10.2.2.8 *Emery paper,* of 0 or 00 grade, or steel wool.

10.2.2.9 *Thermometer,* readable to 1 °C.

10.2.2.10 *Balance,* suitable for determining the sample mass to within 0.1 %.

10.2.2.11 *Means of measuring the sample dimensions,* such as a steel rule graduated to 0.5 mm.

10.2.2.12 *Test sieve,* 10 mm aperture size.

10.2.2.13 *Means of compacting the soil into the test container.* This shall consist of one of the following, depending on the degree of compaction required:

a) a 2.5 kg rammer, as specified in **3.3.2.2** of BS 1377-4:1990;

b) a 4.5 kg rammer, as specified in **3.5.2.2** of BS 1377-4:1990;

c) a vibrating hammer with tamping foot, as specified in **3.7.2.2** and **3.7.2.3** of BS 1377-4:1990.

10.2.2.14 *Large metal tray,* e.g. 900 mm square and 80 mm deep.

10.2.2.15 *Large metal scoop.*

10.2.2.16 *Shredding implement for stiff clays.*

10.2.2.17 *Watertight containers,* such as strong polyethylene bags.

10.2.3 *Procedure*

10.2.3.1 *Preparation of undisturbed sample*

10.2.3.1.1 Prepare the sample and set it up for the test as described in **10.2.3.1.2** to **10.2.3.1.8**. (**10.2.3.1.2** and **10.2.3.1.3** do not apply if the sample is received in a suitable non-conducting container).

10.2.3.1.2 Weigh the test container to the nearest 5 g, and measure its internal diameter D to the nearest 0.5 mm.

10.2.3.1.3 Extrude the sample from the sampling tube, or trim the block sample, and insert the sample into the test container with the minimum of disturbance and loss of moisture. Ensure that the sample is a close fit in the container.

10.2.3.1.4 Trim the ends of the sample to provide smooth flat faces normal to the axis of the container and just inside the ends. Protect the soil from moisture loss.

10.2.3.1.5 Measure the length, L, of the soil sample in the container to the nearest 1 mm by measuring inwards from the ends.

10.2.3.1.6 Weigh the container with sample to the nearest 5 g.

10.2.3.1.7 Clean the contact surfaces of the disc electrodes by rubbing with emery paper or steel wool to give a shiny surface, then wash with clean water and wipe dry with a clean cloth.

NOTE Occasional small patches of corrosion that are difficult to remove are of no consequence.

10.2.3.1.8 Place the electrodes firmly in contact with the soil at either end of the test container, using a light tamping action. Establishment of a good contact is essential.

10.2.3.2 *Preparation of compacted sample of disturbed soil*

10.2.3.2.1 If necessary, remove large particles by passing the soil through a 10 mm test sieve, and spread out the sample on a tray.

10.2.3.2.2 If the moisture content of the soil is to be adjusted, add the appropriate amount of water (see note), or allow the soil to partially dry at room temperature. Mix thoroughly to ensure a uniform distribution of water. Allow a maturing period of at least 16 h for a cohesive soil.

NOTE If possible a sample of the water that will be used on site should be used.

10.2.3.2.3 Take representative samples for the determination of the moisture content, as described in **3.2** of BS 1377-2:1990, to check that the desired moisture content has been achieved.

10.2.3.2.4 Measure the internal diameter, D, of the test container to the nearest 0.5 mm.

10.2.3.2.5 Clean the contact surfaces of the electrodes by rubbing with emery paper or steel wool to give a shiny surface, then wash with clean water and wipe dry with a clean cloth.

NOTE Occasional small patches of corrosion that are difficult to remove are of no consequence.

10.2.3.2.6 Place the bottom electrode in the test container and weigh the assembly to the nearest 5 g.

10.2.3.2.7 Stand the container on a firm flat surface.

10.2.3.2.8 Place the soil in layers into the container and compact each layer by applying the appropriate compactive effort.

NOTE The number of blows applied to each layer to achieve the required density should be determined by trial beforehand.

Compact the final layer with the disc in place.

10.2.3.2.9 Trim the top surface of the sample flat and normal to the axis of the tube and measure the length, L, of compacted soil in the container to the nearest 1 mm.

10.2.3.1.10 Weigh the container assembly and soil to the nearest 5 g.

10.2.3.3 *Measurement of resistivity*

10.2.3.3.1 Connect the terminals on the electrodes to the measuring instrument with the copper wire.

10.2.3.3.2 Apply an initial potential, E (in V) across the electrodes and measure the resulting current I, (in mA or µA).

NOTE. The applied potentials may range from 100 mV to 1 V in increments of 100 mV, and from 5 V to 10 V in steps of 0.5 V, depending on the soil resistivity.

10.2.3.3.3 Repeat step **10.2.3.3.2** with the potential increased by suitable increments for at least five steps.

10.2.3.3.4 Repeat any readings in **10.2.3.3.3** that deviate significantly from the linear relationship when plotted as described in **10.2.4.1**.

10.2.3.3.5 Record to the nearest 1 °C the temperature of the soil immediately after the test.

10.2.3.3.6 Extrude the sample from the container and take at least three representative specimens for the determination of moisture content, as described in **3.2** of BS 1377-2:1990.

10.2.3.3.7 Clean and dry the electrodes before putting away.

10.2.4 ***Plotting and calculation***

NOTE See form 3(g) of Appendix A.

10.2.4.1 Plot each value of voltage potential, E (inV) as ordinate against the reading of current I (in mA or µA) as abscissa, and draw the line of best fit through the plotted points including the origin.

10.2.4.2 Determine the slope of the line $\mathrm{d}E/\mathrm{d}l$ and derive the mean resistance R (in Ω) of the soil sample.

10.2.4.3 Calculate the cross-sectional area A (in mm^2) of the soil sample from the measured diameter D.

10.2.4.4 Calculate the resistivity of the soil, r_s (in Ω·m) from the equation:

$$r_s = \frac{RA}{1000L}$$

where

A is the cross-sectional area of the sample (in mm^2);

L is the length of the sample between the electrodes (in mm);

R is the mean resistance of the soil sample (in Ω).

NOTE Electrical resistivity is usually denoted by the symbol ρ_s, but r_s is used in this standard to avoid confusion with soil particle density.

10.2.4.5 If the test temperature (T °C) was different from 20 °C, calculate the equivalent resistivity at 20 °C, $r_{20,}$ from the equation:

$$r_{20} = \frac{(20 + T)}{40}\, r_s$$

NOTE This correction is valid only for temperatures between 5 °C and 30 °C.

10.2.4.6 Calculate the density of the soil sample ρ (in $\mathrm{Mg/m}^3$) from its dimensions and mass.

10.2.5 *Test report.* The test report shall state that the test was carried out in accordance with **10.2** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the resistivity of the soil sample (in Ω m) to two significant figures, corrected to 20 °C if appropriate;

NOTE If required the corresponding resistivity (in Ω m) in-situ at a temperature of T_1 (in °C) would be equal to:

$$\frac{40}{20 + T_1}\, r_{20}$$

c) the dimensions of the sample tested;

d) the method of compaction of the test sample if relevant, including the number of layers and compactive effort applied to each layer;

e) the relationship of the sample dry density and moisture content to the relevant moisture-density relationship curve for a compacted sample;

f) the type of electrodes used, including material;

g) the temperature at which the test was performed;

h) the moisture content and density of the sample;

i) the ground water level relative to the level from which the sample was taken in-situ;

j) the weather conditions on site at the time of sampling, and the prevailing conditions during the preceding period insofar as they could affect the ground water conditions, if available;

k) the information required by **9.1** of BS 1377-1:1990.

10.3 Measurement of resistivity: Wenner probe method

10.3.1 *General.* This procedure enables the electrical resistivity of an undisturbed or disturbed sample of soil to be measured in the laboratory by a method which is the same in principle as that used in the field (see **5.1** of BS 1377-9:1990).

The test is suitable for samples of the same types as referred to in **10.2.1**, but is not recommended for soils containing an appreciable amount of gravel-size particles.

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

10.3.2 ***Apparatus***

10.3.2.1 *Rigid container* (referred to as the test container) of non-conducting material at least 100 mm across internally and 400 mm to 1 000 mm long. The sampling tube may be used if it is of suitable material.

NOTE Rigid plastics piping of PVC or polypropylene have been found to be suitable.

Clearance holes shall be drilled in the side of the container for insertion of the probes specified in **10.3.2.3**.

Alternatively, a suitable means of supporting the test sample, and a wrapping material such as polyethylene sheet to prevent loss of moisture, is acceptable for cohesive soils.

10.3.2.2 *Suitable means of extruding the sample,* from the sampling tube and into the test container, such as that referred to in **8.2.2** of BS 1377-1:1990. (This is used when an undisturbed sample is not received in a suitable non-conducting container).

10.3.2.3 *Four steel probe electrodes* of equal length with pointed ends in alignment and equally spaced [the Wenner configuration (see Figure 3)].

NOTE Probes 2 mm to 6 mm diameter about 25 mm apart have been found to be satisfactory in clay soils.

10.3.2.4 *Calibrated impedence ohmmeter,* providing an oscillating electricity supply of a frequency between 70 Hz and 200 Hz, preferably avoiding 50 Hz and 60 Hz and their multiples. The total measuring range shall be up to at least 1 500 Ω.

The instrument shall have a performance complying with class **2.5** of BS 89, and shall be calibrated at intervals not exceeding 2 years for common usage.

10.3.2.5 *Apparatus,* as specified in **10.2.2.5** to **10.2.2.11** with the addition of the apparatus specified in **10.2.2.12** to **10.2.2.17** if a compacted sample is to be prepared.

10.3.3 ***Procedure***

10.3.3.1 *Preparation of undisturbed sample*

10.3.3.1.1 Prepare the sample and set it up for the test as described in **10.3.3.1.2** to **10.3.3.1.6** (**10.3.3.1.2** and **10.3.3.1.3** do not apply if the sample is received in a suitable non-conducting container.)

10.3.3.1.2 Weigh the test container to the nearest 5 g, and measure its internal dimensions to the nearest 0.5 mm.

10.3.3.1.3 Extrude the sample from the sampling tube, or trim the block sample, and insert the sample into the test container with the minimum of disturbance and loss of moisture. Ensure that the sample is a close fit in the container. Alternatively tightly wrap the sample in polythene sheet.

10.3.3.1.4 Trim the ends of the sample to provide smooth flat faces normal to the axis of the container and just inside the ends. Protect the soil from moisture loss.

10.3.3.1.5 Measure the length, L, of the soil sample in the container to the nearest 1 mm by measuring inwards from the ends.

10.3.3.1.6 Weigh the container with sample to the nearest 5 g.

10.3.3.2 *Preparation of compacted sample*

10.3.3.2.1 If necessary, remove large particles by passing the soil through a 10 mm sieve, and spread out the sample on a tray.

10.3.3.2.2 If the moisture content of the soil is to be adjusted, add the appropriate amount of water (see note), or allow the soil to partially dry at room temperature. Mix thoroughly to ensure a uniform distribution of water. Allow a maturing period of at least 16 h for a cohesive soil.

NOTE If possible a sample of the water that will be used on site should be used.

10.3.3.2.3 Take a representative sample for the determination of the moisture content, as described in **3.2** of BS 1377-2:1990, to check that the desired moisture content has been achieved.

10.3.3.2.4 Measure the internal dimensions, D, of the test container to the nearest 0.5 mm.

10.3.3.2.5 Stand the container on a firm flat surface, place the soil in layers into the container and compact each layer by applying the appropriate compactive effort.

NOTE The number of blows applied to each layer to achieve the required density should be determined by trial beforehand.

10.3.3.2.6 Trim the top surface of the sample flat and normal to the axis of the tube and measure the length, L, of compacted soil to the nearest 1 mm.

10.3.3.2.7 Weigh the container assembly and soil to the nearest 5 g.

10.3.3.3 *Measurement of resistivity*

10.3.3.3.1 Clean the probes by rubbing with emery paper or steel wool to give a shiny surface, then wash with clean water and wipe dry with a clean cloth.

10.3.3.3.2 Insert the four probe electrodes firmly into the soil sample so that they are equally spaced (see Figure 3). When inserted into the side of a cylindrical sample insert the probes to a depth of at least half, and up to three-quarters, of the diameter of the tube. If a plastics container is being used drill it for inserting the probes. If the sample is wrapped in polythene sheet, jab the probes in so as to cause minimum sample disturbance.

10.3.3.3.3 Connect the terminals on the two outer electrodes to the source of current, and those on the inner two electrodes to the potential terminals on the ohmmeter. The circuit is shown in Figure 3. Make adjustments in accordance with the manufacturer's instructions.

10.3.3.3.4 Pass a stable a.c. current from one outer electrode to the other, operating the ohmmeter in accordance with the manufacturer's instructions.

NOTE A current of about 10 mA is suitable for many soils, but for soils of very high resistivity the impressed voltage might have to be increased.

Some adjustment of the current may be necessary before taking a reading.

10.3.3.3.5 When the instrument shows that the circuit is balanced and stable, read and record the measured resistance between the inner electodes.

10.3.3.3.6 Remove the electrode probes and repeat **10.3.3.3.2** to **10.3.3.3.5** at the other end of the sample, or at a different location spaced at least 50 mm from the first.

10.3.3.3.7 Record the temperature of the soil immediately after the test.

10.3.3.3.8 Extrude the sample from the container and take at least three representative specimens for the determination of moisture content, as described in **3.2** of BS 1377-2:1990.

10.3.3.3.9 Clean and dry the probes before putting away.

10.3.4 *Calculations*

NOTE See form 3(h) of Appendix A.

10.3.4.1 Average the two measured values of the resistance, R (in Ω).

10.3.4.2 Calculate the resistivity of the soil, r_s (in Ω·m) from the equation.

$$r_s = \frac{2\pi aR}{1000}$$

where

a is the centre to centre distance between adjacent electrodes (in mm);

R is the average measured resistance (in Ω).

NOTE Electrical resistivity is usually denoted by the symbol ρ_s, but r_s is used in this standard to avoid confusion with soil particle density.

10.3.4.3 If the test temperature T (in °C) was different from 20 °C, calculate the equivalent resistivity at 20 °C, r_{20}, (in Ω.m) from the equation:

$$r_{20} = \frac{(20 + T)}{40} r_s$$

NOTE This correction is valid only for temperatures between 5 °C and 30 °C.

10.3.5.4 Calculate the density of the soil sample ρ (in Mg/m^3) from its dimensions and mass.

10.3.6 *Test report.* The test report shall state that the test was carried out in accordance with **10.3** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the resistivity of the soil sample (in Ω.m) to two significant figures, corrected to 20 °C if appropriate;

NOTE If required the corresponding resistivity (in Ω.m) in-situ at a temperature of T_1 (in °C) would be equal to:

$$\frac{40}{20 + T_1} r_{20}$$

c) the dimensions of the sample tested;

d) the method of compaction of the test sample if relevant, including the number of layers and compactive effort applied to each layer;

e) the relationship of the sample dry density and moisture content to the relevant moisture-density relationship curve for a compacted sample;

f) the dimensions of probes, their material, spacing and penetration into the soil sample;

g) whether the probes were inserted at the ends or into the side of the sample;

h) the temperature at which the test was performed;

i) the moisture content and density of the sample;

j) the ground water level relative to the level from which the sample was taken in-situ;

k) the weather conditions on site at the time of the sampling, and the prevailing conditions during the preceding period insofar as they could affect the ground water conditions, if available;

l) the information required by **9.1** of BS 1377-1:1990.

10.4 Measurement of resistivity: open container method

10.4.1 *General.* This procedure is for the measurement of the electrical resistivity of a soil such as fill material that has been compacted to a specified density and then saturated by adding water. It is suitable only for relatively free-draining materials into which water can percolate easily.

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

10.4.2 ***Apparatus***

10.4.2.1 *Rectangular box with transparent sides,* of internal dimensions approximately 230 mm long, 100 mm wide and 100 mm high, of non-conductive material, fitted with 100 mm^2 electrodes of stainless steel set at about 15 mm from each end of the box. Small holes in the electrodes allow passage of water. The electrodes are fitted with terminals for connecting insulated copper wire. The sides of the box are of suitable non-conducting material.

The general arrangement is indicated in Figure 4.

10.4.2.2 *Calibrated Kohlrausch null-deflection bridge,* suitable for measuring electrical resistances in the range 10 Ω to 1 MΩ to an accuracy of 5 %, which provides alternating current output at 1 000 Hz.

The instrument shall be re-calibrated at intervals not exceeding 12 months.

10.4.2.3 *Insulated copper wire,* for connecting the electrodes to the resistivity meter, fitted at each end with suitable terminals to ensure good electrical contact.

NOTE Standard copper wire of about 1.5 mm^2 cross-sectional area is suitable.

10.4.2.4 *Test sieve,* 10 mm aperture size.

10.4.2.5 *Steel tamping bar.* One with a 20 mm^2 tamping end, weighing about 0.6 kg, has been found suitable.

10.4.2.6 *Thermometer,* readable to 1 °C.

10.4.2.7 1L *measuring cylinder.*

10.4.2.8 *Balance,* readable to 5 g.

10.4.2.9 *Large metal tray,* e.g. 900 mm square and 80 mm deep.

10.4.2.10 *Large metal scoop.*

10.4.2.11 *Small tools such as a spatula and garden trowel.*

10.4.2.12 *Steel straightedge,* about 150 mm long.

10.4.2.13 *Watertight containers,* such as strong polyethylene bags.

10.4.2.14 *Apparatus for the determination of moisture content,* as described in **3.2.2**. of BS 1377-2:1990.

10.4.2.15 *Emery paper,* of 0 or 00 grade.

10.4.2.16 *Means of measuring the sample dimensions,* such as a steel rule graduated to 1 mm.

10.4.3 ***Procedure***

10.4.3.1 *Preparation of sample*

10.4.3.1.1 Clean the contact surfaces of the electrodes by rubbing with emery paper to give a shiny surface, then wash with clean water and wipe dry with a clean cloth.

NOTE Occasional small patches of corrosion that are difficult to remove are of no consequence.

10.4.3.1.2 Measure the internal dimensions of the soil compartment of the box to the nearest 1 mm.

10.4.3.1.3 If necessary, remove large particles by passing the soil through a 10 mm test sieve. Determine the percentage by mass of particles removed.

10.4.3.1.4 Weigh out, to the nearest 5 g, a mass of soil in excess of the amount required to fill the box.

10.4.3.1.5 Take representative specimens of the soil for determination of the moisture content, in accordance with **3.2** of BS 1377-2:1990.

10.4.3.1.6 Place the soil into the box in three equal layers and compact each layer with 30 similar blows of the tamping bar.

NOTE If required the soil may be compacted to achieve a specified density, in which case the number and intensity of blows applied to each layer should be determined by trial beforehand.

10.4.3.1.7 Trim the top surface of the soil level and flush with the top edge of the box, using fine material to fill any voids.

10.4.3.1.8 Retain and weigh all the material not compacted into the box, including moisture content specimens, and obtain the mass of soil used by difference.

10.4.3.2 *Measurement of resistivity*

10.4.3.2.1 Saturate the soil by pouring water from a filled measuring cylinder on to the soil and into the water chambers until all chambers are completely filled.

NOTE Distilled water should be used unless the material in-situ will be below the water table, in which case water from the site should be used.

10.4.3.2.2 Record the volume of water remaining in the measuring cylinder and calculate by difference the volume of water used.

10.4.3.2.3 Connect the electrodes without delay to the Kohlrausch bridge with the copper wire and apply a potential difference across the electrodes.

10.4.3.2.4 Balance the bridge and determine the resistance, R (in Ω) of the soil between the two electrodes.

10.4.3.2.5 Repeat step **10.4.3.2.4** after allowing the soil to stand for 1 h after saturation, and topping up with water if necessary. Measure the temperature of the water.

NOTE Alternating current will cause a temperature rise if continued for a length of time.

10.4.3.2.6 Record the temperature of the soil.

10.4.3.2.7 Clean and dry the electrodes after removing the soil and water from the box.

10.4.4 *Calculations*

NOTE See form 3(i) of Appendix A.

10.4.4.1 For each measurement of resistance calculate the resistivity of the soil, r_s (in Ω·m) from the equation:

$$r_s = \frac{RA}{1000L}$$

where

R is the measured resistance between the electrodes (in Ω);

A is the cross-sectional area of the soil chamber (in mm^2);

L is the length of the soil chamber between the electrodes (in mm).

NOTE Electrical resistivity is usually denoted by the symbol ρ_s, but r_s is used in this standard to avoid confusion with soil particle density.

10.4.4.2 If the test temperature T (in °C) was different from 20 °C, calculate the equivalent resistivity at 20 °C, r_{20}, (in Ω·m) from the equation:

$$r_{20} = \frac{20 + T}{40} r_s$$

NOTE This correction is valid only when the temperature T lies between 5 °C and 30 °C.

10.4.4.3 Calculate the volume of water contained in the two chambers behind the electrodes.

10.4.4.4 Calculate the volume of water required to saturate the soil by deducting the above from the total volume of water added to saturate the soil.

10.4.4.5 Calculate the density of the soil as compacted into the box, from its mass and volume.

10.4.5 *Test report.* The test report shall state that the test was carried out in accordance with **10.4** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the resistivity of the saturated soil sample initially, and after 1 h of saturation (in Ω·m), to two significant figures, corrected to 20 °C if appropriate;

NOTE If required the corresponding resistivity (in Ω·m) in-situ at a temperature of T_1 (in °C) would be equal to:

$$\frac{40}{20 + T_1} r_{20}$$

c) the test temperature (in °C);

d) the density to which the soil was compacted and its mosture content;

e) the dimensions of the sample;

f) the volume of water required to saturate the soil;

g) the approximate percentage of large particles removed from the original sample, if any;

h) the information required by **9.1** of BS 1377-1:1990.

11 Determination of the redox potential

11.1 General

11.1.1 *Principle.* The test described in this clause is for the determination of the redox potential of a sample of soil in the laboratory.

The method used is to measure the electro-chemical potential between a platinum electrode and a saturated calomel reference electrode in contact with the soil.

NOTE Other types of reference electrode are available but calomel is generally the most satisfactory.

The pH of the soil also has to be determined to enable the standardized redox potential to be calculated.

11.1.2 *Purpose.* The redox potential provides a means of assessing whether a soil is conducive to the activity of sulphate-reducing bacteria, which cause corrosion of metals. These bacteria, which are present in most soils, flourish under reducing conditions (low redox potential) and become dormant under oxidizing conditions (high redox potential).

Changes in soil properties can cause enhanced corrosion where continuous buried metals, e.g. steel pipelines, pass from one soil type to another.

11.1.3 *Limitations.* Redox potential results are of significance only for soil samples that have not been disturbed. Tests on soil that has been recompacted in the laboratory are of questionable value because of the change in properties resulting from unavoidable exposure to the atmosphere.

Laboratory tests do not take account of changes in the soil characteristics which take place with time. These can be assessed only by carrying out in-situ tests at appropriate intervals, as described in BS 1377-9:1990.

Results of these tests should be interpreted by a specialist.

11.1.4 *Type of sample.* The undisturbed sample used for the test may be obtained in a sampling tube from a borehole, or prepared from a block sample taken from an open excavation, provided that it can be adequately sealed to prevent atmospheric oxidation. Samples of cohesionless soil should be taken in a non-conducting container which can be adequately sealed and which is suitable for the test.

NOTE Metal containers can cause changes in the redox value with time.

Laboratory redox tests should be carried out immediately after sampling.

The requirements of Part 1 of this standard, where appropriate, shall apply to this test method.

11.2 Apparatus

11.2.1 *Rigid container,* (referred to as the test container) of suitable non-conducting material at least 100 mm across internally and 400 mm to 1 000 mm long. The sampling tube may be used if it is of suitable material.

NOTE Rigid plastics piping of PVC or polypropylene have been found to be suitable.

11.2.2 *Suitable means of extruding the undisturbed sample,* from the sampling tube and into the test container (when the sample is not received in a suitable non-conducting container), such as that referred to in **8.2.2** of BS 1377-1:1990.

11.2.3 *Platinum probe,* having two separate platinum electrodes embedded in the nosepiece, with a means of protection when not in use. The probe is provided with a connecting lead permitting the inclusion of each platinum electrode individually in an electric circuit, and each connection is separately identified.

11.2.4 *Calomel reference probe,* having a mercury/mercurous chloride reference electrode which can be refilled and with a connection to a ceramic junction. The calomel reference electrode shall be kept clean when not in use by being stored in a sealed container. When not in use, precipitation of crystals due to evaporation loss shall be prevented, particularly at the porous junction, by storing upright and closing the breather hole, or by immersion in saturated potassium chloride solution.

NOTE The platinum and calomel probes are often separate and made of glass and are therefore fragile.

11.2.5 *Calibrated voltmeter,* having a performance complying with class **2.5** of BS 89. The total measuring range shall be 0 V d.c. to 2 V d.c. with a readability within 10 mV. The input impedance shall be not less than 10^6 Ω and the polarity (positive or negative) shall be marked on the two input terminals.

The instrument shall be re-calibrated at intervals not exceeding 12 months.

11.2.6 *Insulated flexible electric cable and connectors,* for use with the probes.

11.2.7 Apparatus for measuring the pH value of soil, as described in **9.2**.

11.2.8 *Thermometer,* readable to 1 °C.

11.2.9 *Apparatus for the determination of moisture content,* as described in **3.2.2** of BS 1377-2:1990.

11.2.10 *Sample trimming tools,* e.g. trimming knife, spatula, end-trimming tool.

11.2.11 *Paper tissues*

11.2.12 *Absorbent surgical cotton-wool swabs*

11.3 Reagents

11.3.1 *Potassium chloride,* saturated solution contained in a screw-topped plastics bottle with a pouring lip, suitable for filling the reservoir of the calomel reference probe, or with a separate small dropper or syringe. 500 mL is a suitable quantity.

11.3.2 *Jeweller's rouge*

11.3.3 *Colourless methylated spirit,* about 70 % strength with 30 % distilled water, in a screw-topped wide-mouth bottle. 500 mL is a suitable quantity.

11.3.4 *Distilled water,* in two differently marked wash bottles, for cleaning platinum electrodes. 500 mL is a suitable quantity for each bottle.

11.4 Procedure

11.4.1 *Preparation of sample.* Test an undisturbed sample taken in a non-conducting container in the same container. Otherwise extrude the sample and place it in a container of suitable non-conducting material for the test with the minimum of exposure to air.

A block or lump sample, wrapped tightly in polythene sheet immediately after taking from the ground, is also suitable and need not be transferred to another container.

Test the sample within 2 days of sampling. Keep exposure to air to a minimum.

11.4.2 *Preparation of probes*

11.4.2.1 Keep the probes upright to prevent leakage of the solution.

11.4.2.2 Assemble the calomel reference probe in accordance with the operating instructions, ensuring that the unit is full of a saturated solution of potassium chloride and that the solution moistens the porous plug.

11.4.2.3 Remove any air bubbles in the potassium choride solution by gently tapping the probe, and remove excess fluid from the porous plug.

11.4.2.4 Clean and polish the platinum electrodes as necessary. Initially smear the surface lightly with moist jeweller's rouge and use gently abrasive action with cotton-wool swabs.

11.4.2.5 Wash, using the 70 % methylated spirit.

11.4.2.6 Wash thoroughly with distilled water.

NOTE The electrodes are washed by dipping into first one and then the other bottle of distilled water. The different markings on the bottles help ensure that the same sequence is always followed.

11.4.2.7 Dry each electrode with clean paper tissues.

11.4.2.8 Assemble the probes ready for insertion into the soil sample.

11.4.2.9 Connect the cable from the positive terminal of the voltmeter to one of the platinum electrodes and the cable from the negative terminals to the calomel reference electrode, but leave the circuit open. This circuit is considered to give positive readings.

11.4.3 *Measurement of redox potential*

11.4.3.1 Insert the probes into the soil sample about 100 mm apart. Ensure that the platinum probe penetrates to a depth of at least 50 mm.

11.4.3.2 Rotate the platinum probe by about a quarter up to one turn without allowing air to reach the probes to ensure good contact with the soil.

11.4.3.3 Close the electric circuit and record the voltage reading to the nearest 10 mV, and its polarity (positive or negative), when the voltage becomes steady.

NOTE Occasionally the current between the platinum electrode and the reference electrode will be in the reverse direction such as to require the connections to the millivoltmeter to be reversed. In this case the reading should be considered to be negative.

11.4.3.4 Transfer the connection from the positive terminal of the voltmeter to the other platinum electrode.

11.4.3.5 Repeat steps **11.4.3.2** and **11.4.3.3**.

11.4.3.6 If the two readings differ by more than 20 mV remove the probes, re-clean the platinum electrodes and re-install in a different position at least 50 mm away, or at the other end, pushing the probes further into the soil if possible. Do not re-install in the orignal positions as oxygen will have penetrated and a false reading could result.

11.4.3.7 Repeat steps **11.4.3.2** to **11.4.3.5**. If the two readings again differ by more than 20 mV and there is not enough length of sample for another repeat test, reject the sample, clean the electrodes and perform the test on another sample of the same soil.

11.4.3.8 If there is a sufficient length of sample, obtain another set of readings as described in **11.4.3.2** to **11.4.3.7**.

11.4.3.9 Remove the probes, clean the electrodes as described in **11.4.2.4** to **11.4.2.7** and store them as described in **11.2.4**.

11.4.3.10 Record the temperature of the soil to the nearest 1 °C.

11.4.3.11 Take a sample of the soil from the zone of the test and place it in a sealed container.

NOTE If a sample is required for microbiological examination the container should have been cleaned and sterilized beforehand and should be of about 500 mL capacity. Fill the container completely to minimize the air voids.

11.4.3.12 Use a portion of the sample for the determination of moisture content, as described in **3.2** of BS 1377-2:1990.

11.4.3.13 Determine the pH of the sample, in accordance with the procedure described in **9.5**.

11.5 Calculations

11.5.1 Calculate the mean of the acceptable voltage readings and record this value (positive or negative) as the potential of the platinum probe E_p to the nearest 10 mV.

11.5.2 Calculate the redox potential, E_h (in mV), from the equation:

$$E_h = E_p + 250 + 60\,(\mathrm{pH} - 7)$$

where

E_p is as observed above, and may be a positive or negative value;

pH is the pH value of the aqueous suspension of soil determined in accordance with **9.5**.

The constant 250 is the correction factor for a calomel reference probe for converting the measurement to that of a standard hydrogen electrode.

NOTE For other types of reference probe, different constants are required. (see **5.2.1** of BS 1377-9:1990).

11.6 Test report

The test report shall state that the test was carried out in accordance with clause **11** of BS 1377-3:1990 and shall contain the following information:

a) the method of test used;

b) the mean value of the potential (in mV) of the two platinum probes, to the nearest 10 mV;

c) the redox potential (in mV) to the nearest 10 mV;

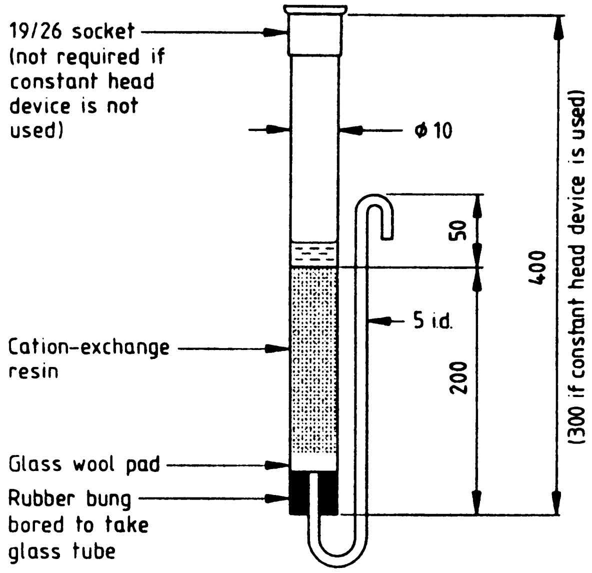
d) the pH value of the soil suspension;

e) the type of reference probe;

f) the type and approximate size of the sample;

g) the temperature of the sample at the time of test;

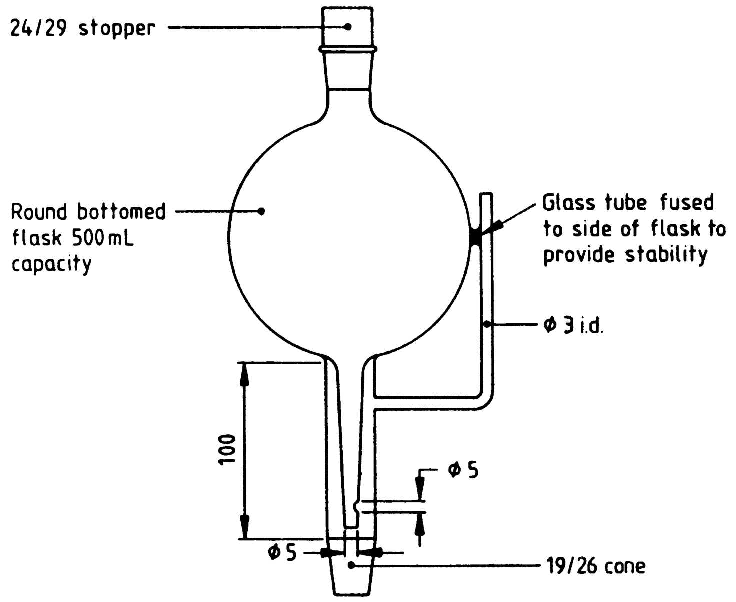
h) the information required by **9.1** of BS 1377-1:1990.



All dimensions are in millimetres.

NOTE. See **4.1.3.1.1** of BS 1377 : Part 1 : 1990.

Figure 1 — Ion-exchange column for sulphate determination



All dimensions are in millimetres.

NOTE. See 4.1.3.1.1 of BS 1377 : Part 1 : 1990.

Figure 2 — Constant-head device for use with ion-exchange column

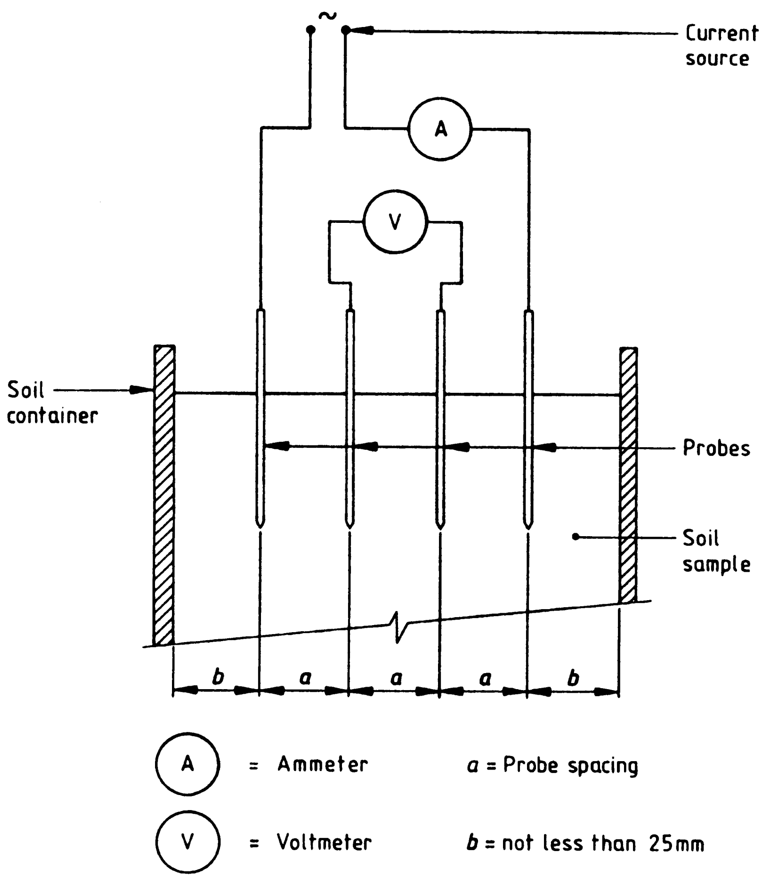
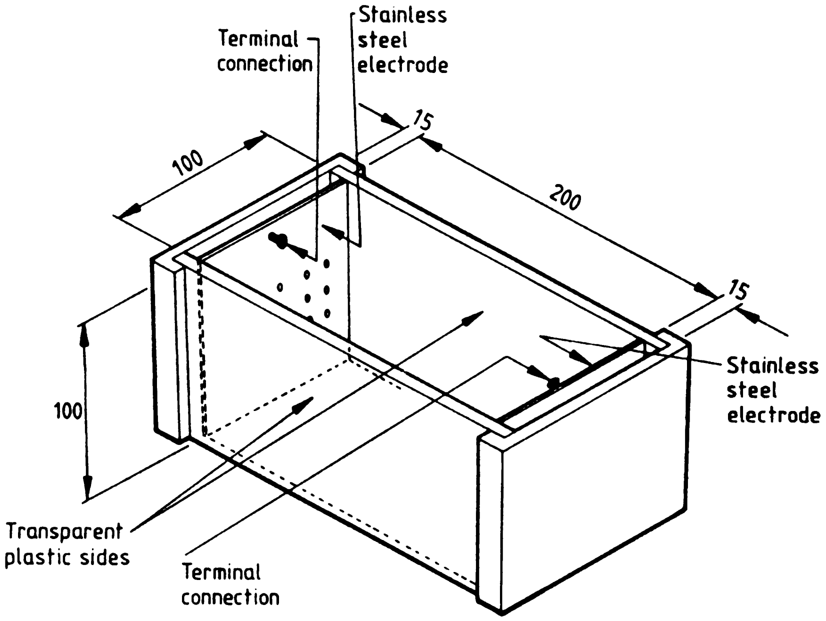


Figure 3 — Circuit diagram for resistivity test using Wenner probes



All dimensions are in millimetres.

NOTE. See **4.1.3.1.1** of BS 1377 : Part 1 : 1990.

Figure 4 — Container for resistivity tests on saturated granular soil

Appendix A Typical test data and calculation forms

The test sheets shown below are given as examples only; other suitable forms may be used.

Form 3(a) Organic matter content
Form 3(b) Loss on ignition
Form 3(c) Sulphate content: gravimetric method
Form 3(d) Sulphate content: ion-exchange method
Form 3(e) Carbonate content: rapid titration method
Form 3(f) Water-soluble/acid-soluble chlorides
Form 3(g) Electrical resistivity: disc electrode method
Form 3(h) Electrical resistivity: Wenner probe method
Form 3(i) Electrical resistivity: open container method

Form 3(a). Organic matter content							
Location						Job ref.	
						Borehole/ Pit no.	
Soil description						Sample no.	
						Depth	m
						Date	
Test method						BS 1377-3:1990:**3.4**	
Specimen reference							
Mass of original sample m_1	g						
Mass of soil passing 2 mm test sieve m_2	g						
Weighing bottle number							
Mass of weighing bottle and dry soil before taking test specimen	g						
Mass of weighing bottle and dry soil after taking test specimen	g						
Mass of dry soil used m_3	g						
Volume of ferrous sulphate solution added to standardize potassium dichromate solution x	ml						
Volume of ferrous sulphate solution used to oxidize excess potassium dichromate solution y	ml						
Volume of potassium dichromate solution used to oxidize organic matter in soil $V = 10.5(1-y/x)$	ml						
Percentage of organic matter in soil specimen finer than 2 mm $\frac{0.67V}{m_3}$	%						
Percentage finer than 2 mm in original sample $\frac{m_2}{m_1} \times 100$							
Remarks:		Operator		Checked		Approved	

Form 3(b). Loss on ignition

Location	Job ref.	
	Borehole/ Pit no.	
Soil description	Sample no.	
	Depth	m
	Date	

Test method BS 1377-3:1990:**4.3**

Specimen reference					
Mass of initial sample m_1	g				
Mass of soil passing 2 mm test sieve m_2	g				
Crucible no.					
Mass of crucible m_c	g				
Mass of crucible and dry soil m_3	g				
Mass of crucible and soil after ignition m_4	g				
Loss on ignition, L0I, as percentage of soil finer than 2 mm $\frac{m_3 - m_4}{m_3 - m_c} \times 100$	%				
Percentage finer than 2 mm in original sample $\frac{m_2}{m_1} \times 100$	%				

Remarks:	Operator	Checked	Approved

Form 3(c). Sulphate content: gravimetric method
Samples of soil/ground water[a]

Location	Job ref.	
	Borehole/ Pit no.	
Soil description	Sample no.	
	Depth	m
	Date	

Test method BS 1377-3:1990:**5.5**

Specimen reference							
Mass of original sample m_1	g						
Mass of soil passing 2 mm test sieve m_2	g						
Percentage finer than 2 mm in original sample $\frac{m_2}{m_1} \times 100$	%						
Weighing bottle number							
Mass of weighing bottle and oven dry soil before taking test specimen	g						
Mass of weighing bottle and oven dry soil after taking test specimen	g						
Mass of dry soil used m_3	g						
Crucible number							
Mass of crucible and ignited precipitate	g						
Mass of crucible	g						
Mass of ignited precipitate (first weighing)	g						
Mass of ignited precipitate (second weighing) m_4	g						
Soil samples: acid extract[a] Percentage of total sulphates (as $S0_3$) in soil specimen finer than 2 mm $\frac{34.3 m_4}{m_3}$	%						
Soil samples: water extract[a] Percentage of water-soluble sulphate (as SO_3) in soil specimen finer than 2 mm $1.372\ m_4$	%						
Ground water samples[a] Sulphate content of ground-water (as $S0_3$) $6.86\ m_4$	g/L						

[a] Delete as appropriate.

Remarks:	Operator	Checked	Approved

Form 3(d). Sulphate content: ion-exchange method
Samples of soil/ground water[a]

Location	Job ref.	
	Borehole/ Pit no.	
Soil description	Sample no.	
	Depth	m
	Date	
Test method	BS 1377-3:1990:**5.6**	

Specimen reference							
Ground water (GW) or soil (SS) sample							
Initial mass of sample m_1[a]	g						
Mass of soil sample passing 2 mm test sieve m_2[a]	g						
Percentage finer than 2 mm in original sample[a] $\frac{m_2}{m_1} \times 100$	%						
Mass of watch glass and soil[a]	g						
Mass of watch glass[a]	g						
Mass of soil used[a]	g						
Volume of NaOH in burette before titration	mL						
Volume of NaOH in burette after titration	mL						
Volume of NaOH used V	mL						
Concentration of NaOH solution B	mol/L						
Sulphate content of water-soil extract[a] $SO_3 = 0.8\,BV$ or $SO_3 = 0.16\,BV$	g/L[a] %[a]						
Sulphate content of ground water $SO_3 = 0.4\,BV$[a]	g/L						

[a] Delete as appropriate.

Remarks:	Operator	Checked	Approved

From 3(e). Carbonate content: rapid titration method

Location	Job ref.	
	Borehole/ Pit no.	
Soil description	Sample no.	
	Depth	m
	Date	
Test method	BS 1377-3:1990:**6.3**	

Specimen reference								
Mass of dry specimen m		g						
Volume of acid used standardizing HC1 solution	(1)	mL						
	(2)	mL						
	(3)	mL						
Mean volume of acid used V_1		mL						
Concentration of acid solution H $H = \frac{V_1}{25B}$								
Volume of sodium hydroxide solution used V_2		ml						
Carbonate content of soil, CO_2 $\frac{8.8(25H - BV_2)}{m}$		%						

Remarks:	Operator	Checked	Approved

From 3(f). Chloride content
Water-soluble/acid soluble[a] chlorides

Location	Job ref.	
	Borehole/ Pit no.	
Soil description	Sample no.	
	Depth	m
	Date	
Test method	BS 1377-3:1990:**7.2/7.3**[a]	

Specimen reference							
Water extract only[a]							
Mass of bottle	g						
Mass of bottle and dry soil	g						
Mass of dry soil m	g						
Mass of distilled water added m_w	g						
Water: soil ratio $r = \frac{m_w}{m}$							
Volume of thiocyanate solution used for standardization V_1	mL						
Concentration of thiocyanate solution $C = \frac{2.5}{V_1}$	mol/L						
Volume of silver nitrate used V_2	mL						
Volume of thiocyanate solution used V_3	mL						
Water-soluble chloride ion content[a] $0.007092\ (V_2 - 10C\ V_3)$	%						
Acid-soluble chloride ion content[a] $0.07092\ (V_2 - 10C\ V_3)$	%						

[a] Delete as appropriate.

Remarks:	Operator	Checked	Approved

From 3(g). Electrical resistivity: Disc electrodes method

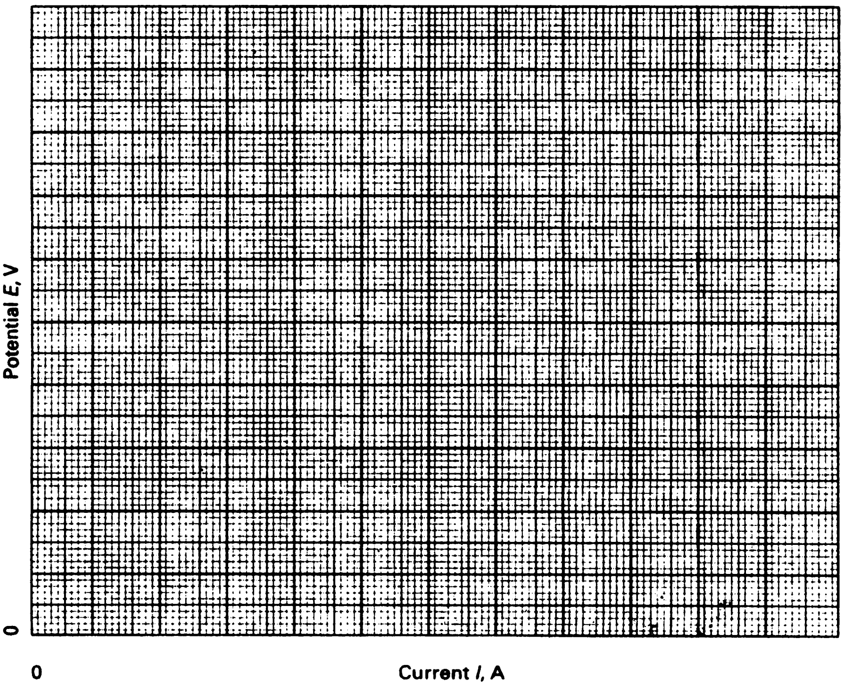
Location	Job ref.	
	Borehole/ Pit no.	
Soil description	Sample no.	
	Depth	m
	Date	

Test method BS 1377-3:1990:**10.2**

Undisturbed/Compacted[a] sample			Compaction procedure[a]		
Sample details					
Mass of container	g		Length of sample	mm	
Mass of container and soil	g		Internal diameter of container	mm	
Mass of soil	g		Cross-sectional area A	mm^2	
Density of sample	Mg/m^3		Volume of sample	cm^3	
Length between electrodes L	mm		Test temperature T	°C	

Resistivity readings

Potential E V	Current I A



From slope of graph, mean resistance = Ω

Soil resistivity $r_s = \dfrac{RA}{1000L}$	Ω m	Moisture content %
Resistivity corrected to 20 °C $r_{20} = \dfrac{(20 + T)\ r_s}{40}$	Ω m	

[a] Delete as appropriate.

Remarks:	Operator	Checked	Approved

Form 3(h). Electrical resistivity: Wenner probe method

Location	Job ref.	
	Borehole/ Pit no.	
Soil description	Sample no.	
	Depth	m
	Date	
Test method		BS 1377-3:1990:**10.3**

Undisturbed/Compacted[a] sample			Compaction procedure[a]		
Sample details					
Mass of container	g		Length of sample L	mm	
Mass of container and soil	g		Internal diameter of container D	mm	
Mass of soil	g		Cross-section area (A)	mm^2	
Density of sample	Mg/m^3		Volume of sample	cm^3	
Spacing between adjacent electrodes a	mm		Test temperature (T)	°C	
Penetration of electrodes	mm				
Resistance readings			Sketch showing locations of electrodes		
Resistance at location (1)	Ω				
Resistance at location (2)	Ω				
Average resistance R	Ω				

Soil resistivity $r_s = \frac{2\,aR}{1000}$	Ω·m	Moisture content %
Resistivity corrected to 20 °C $r_{20} = \frac{(20 + T)\,r_s}{40}$	Ω·m	

[a] Delete as appropriate

Remarks:	Operator	Checked	Approved

Form 3(i). Electrical resistivity: open container method

Location		Job ref.	
		Borehole/ Pit no.	
Soil description		Sample no.	
		Depth	m
		Date	
Test method	BS 1377-3:1990:**10.4**		

Sample details		
Initial mass of soil	g	
Mass retained on 10 mm test sieve	g	
Mass of soil taken	g	
Mass for moisture content	g	
Mass remaining	g	
Total mass not used	g	
Mass of soil for test	g	
Density of test sample	Mg/m^3	
Moisture content	%	
Dry density	Mg/m^3	

Container details		
Total length	mm	
Length between electrodes (*L*)	mm	
Width	mm	
Height	mm	
Cross-sectional area (*A*)	mm^2	
Volume of soil	cm^3	
Test temperature *T*	°C	

Addition of water to soil		Initially	After 1 h
Initial volume	mL		
Volume remaining	mL		
Volume added to box	mL		
Volume of outer chambers	mL		
Volume to saturate sample	mL		
Resistance measurements (*R*)	Ω		
Soil resistivity $\frac{RA}{1000L}(r_s)$	Ω m		
Resistivity corrected to 20 °C $r_{20} = \frac{(20 + T)\ r_s}{40}$	Ω m		

Remarks:	Operator	Checked	Approved

Publications referred to

BS 89, *Specification for direct acting indicating electrical measuring instruments and their accessories.*
BS 1047, *Specification for air-cooled blastfurnace slag aggregate for use in construction.*
BS 1377, *Methods of test for soils for civil engineering purposes.*
BS 1377-1, *General requirements and sample preparation.*
BS 1377-2, *Classification tests.*
BS 1377-4, *Compaction-related tests.*
BS 1377-9, *In-situ tests.*
BS 1881, *Testing concrete.*
BS 1881-124, *Methods for analysis of hardened concrete.*
BS 5930, *Code of practice for site investigations.*
DD 175, *Code of practice for the identification of potentially contaminated land and its investigation*[1].

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[1] Referred to in the foreword only.

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